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ANALYSIS OF A SIMPLE SALT

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ANALYSIS OF A SIMPLE SALT.

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TABLES FOR THE ANALYSIS OF A SIMPLE SALT, PRINTED ON STOUT LINEN.

In Active Preparation.

A QUALITATIVE ANALYSIS, INCLUDING THE SEPARATION OF MIXTURES.

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Univ. Corr. Coll. Tutorial Series.

ANALYSIS OF A SIMPLE SALT.

WITH A SELECTION OF MODEL ANALYSES.

BY

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LIST OF REAGENTS AND APPARATUS

PROVIDED FOR THE PRACTICAL EXAMINATIONS IN CHEMISTRY
AT THE UNIVERSITY OF LONDON.

Fluid Reagents.

In 6 oz. Stoppered Bottles.

Sulphuric Acid (pure and dilute).
Nitric Acid (pure and dilute).
Hydrochloric Acid (pure and dilute).

The dilute acids should be made by mixing one part of the pure acid with three parts of water:

Sulphuretted Hydrogen water.
Ammonium Hydrate.
Ammonium Chloride.
Ammonium Sulphide.
Ammonium Carbonate.
Sodium Hydrate
Sodium Carbonate.
Barium Chloride.

In 4 oz. Stoppered Bottles.

Sodium Phosphate.
Potassium Ferrocyanide.
Potassium Ferricyanide.
Potassium Chromate.
Ammonium Oxalate.
Calcium Sulphate.
Ferric Chloride.
Lead Acetate.
Mercuric Chloride.
Stannous Chloride.
Alcohol.

In 2 oz. Stoppered Bottles.

Platinic Chloride.
Silver Nitrate.
Cobalt Nitrate.

In 4 oz. Stoppered Bottles.

⁂ *These reagents are for occasional use as special tests.*

Hydrofluosilicic Acid.
Tartaric Acid.
Starch Paste.
Potassium Iodide.
Potassium Nitrate.
Potassium Cyanide.
Sodium Acetate.
Chloride of Soda.
Magnesium Sulphate.
Calcium Chloride.
Ammonium Molybdate.
Ammonium Sulphate.
Chlorine Water.
Carbon Disulphide.
Acetic Acid.

Dry Reagents.

In 2 oz. Wide-mouthed Corked Bottles.

Fusion Mixture.
Sodium Carbonate.
Borax.
Potassium Nitrate.
Potassium Cyanide.
Ferrous Sulphate.
Calcium Hydrate (slaked lime).
Black Oxide of Manganese.

Apparatus.

Wash-bottle (page 21).
Sulphuretted Hydrogen apparatus (page 28).
(Ferrous Sulphide will be required for this apparatus).
2 doz. test tubes, 6 × ⅝ in.
2 boiling tubes, 8 × 1¼ in.
Test-tube brush.
Test-tube stand to hold 12 at least.
Piece of wire gauze.
Piece of platinum-foil, 2 in. × 1 in.
3 inches platinum wire.
Black's blowpipe.
6 bulb-tubes.
3 watch-glasses.
2 porcelain evaporating basins, 2 oz. and 4 oz.
3 funnels, two 2-in. and one 3-in.
Filtering papers.
Bunsen burner, or 1 spirit lamp, 4 oz.
Pair of brass or iron crucible tongs.
Wedgewood ware mortar and pestle, 4 oz. at least.
½ lb. tubing and rod, various.
Pieces of charcoal for blowpipe work.
Litmus and Turmeric papers.

In the absence of a local vendor of apparatus the address of a large firm, as Messrs. Burgoyne & Co., 16 Coleman Street, London, E.C., may be useful.

Messrs. W. B. Clive & Co., 13 Booksellers Row, Strand, W.C., to meet the requirements of the Inter. Sc. and Prelim. Sci. Examination of London University, keep in stock—

A Set of Apparatus at 10*s*. 6*d*. or 21*s*.,
A Set of Reagents at 21*s*.,
A Selection of suitable Salts for Analysis at 5*s*.,

but do not supply single pieces of apparatus, or chemicals out of their sets.

PREFACE.

Every one who has tried to do practical work in Science with no resources but his own knows too well the difficulties which very frequently crop up, and which, though seemingly insuperable to him, may be cleared up in a minute or two by a teacher.

With very little preliminary ocular demonstration, it is hoped that students will be able to work their way through this book—ability to analyse a simple salt may be derived from it without being involved in the tedious complications which arise from simultaneous methods of analysis of mixtures as set out in most books. Nevertheless, there is nothing in this book to be unlearned if the student proceeds to more advanced work, which, with the good foundation that may be derived from our chapters and tables, should be no difficult task. No attempt has been made to render this a cram book, on the other hand its treatment of the subject is somewhat liberal, and aims at giving the student an intelligent grasp of the principles of the subject. Its size is greater than that of the usual run of elementary books on analysis, but this is mainly due to the detailed explanation so necessary for those who can get little or no assistance from a teacher.

DIRECTIONS.

The student is advised to learn the tests for each metal and acid before performing the experiment, and to regard the one numbered in dark type as of special importance; it is to be used as the confirmation of his inference derived from the systematic analysis described in Chapter IV.

Most people find it interesting to work through the systematic table for each group before proceeding to the next, and this is in no way injurious.

The following should be taken as the golden rules of analysis:—

Use small quantities of the substance to be tested, even if you have a large stock of it at your disposal; otherwise reactions will often be masked, especially those performed in the dry way. If you have only a small allowance, treasure it, and always keep some in reserve to fall back upon.

Work systematically, avoid making random shots, and **record your processes and results** in an orderly way.

Do not assume what you think ought to have happened if it does not; the basis of your expectation may be erroneous, and in laboratory practice should be confirmed, though it is foolish to follow up an unsatisfactory result in an examination.

Make a note of seeming discrepancies of results: you will probably find the explanation later yourself.

Get some one to test you frequently: any one may do this roughly by furnishing you with a numbered test tube or packet, of which the contents is known to them. Remember that it is permissible for an examiner to try your skill on a base *or* an acid alone, or even on *aqua purissima.*

Keep your test tubes and vessels scrupulously clean. Wash them and your platinum wire at the end of your work each day; try the wire alone in the flame before using it.

Be exact in carrying out the directions given.

INTRODUCTION.

Qualitative Analysis has for its object the determination of the number and nature of the constituents of substances without reference to their proportions.

Usually it has reference to the examination of *salts, i.e.*, combinations of *acids* and *bases*, and in such cases the problem involved is the determination of the particular base and acid present in any given substance. Thus we examine substances to determine, not directly what *elements* are present, but what metals and what acid radicles—the latter, of course, being usually groups of elements.

In determining the metal or metals present in a given substance we submit a portion of the substance to the action of various reagents. These reagents are applied in the dry way, *e.g.*, before the blowpipes, or in the wet way, *i.e.*, in the state of solution. The latter method is the more important. Usually, the phenomenon which we take advantage of in detecting any substance is the formation of a *precipitate*, when solutions of certain bodies are brought together. The formation of a precipitate, of course, is due to the interaction of the substances in solution giving rise to the production of some new compound which is insoluble, or only slightly soluble, in water.

Reagents are divided into *Group reagents* and *special reagents.* The former are those which produce a similar general result with several metals—*e.g.*, Hydrochloric acid gives a white precipitate with either silver, mercury, or lead, and is therefore the group reagent for those metals.

Special reagents are those which we make use of for confirming the presence or absence of a certain metal. In applying reagents to a body to be analysed, it is of the utmost importance that they should be used in a systematic order; otherwise altogether erroneous results may be obtained.

The group reagents usuallyemployed are the following:—Hydrochloric acid; Sulphuretted hydrogen in presence of hydrochloric acid; Ammonia in presence of ammonium chloride; Ammonium sulphide in presence of ammonia and ammonium chloride; Ammonium carbonate in presence of ammonia and ammonium chloride, applied in the above order.

The metals are usually divided into six* groups, according to their behaviour with the above reagents.

Group I. comprises those metals whose chlorides are insoluble in water, and which, therefore, are precipitated by hydrochloric acid (or, indeed, by any soluble chloride). They are Silver, Lead, and Mercury(ous).

Group II. comprises *those of the remaining metals* whose sulphides are insoluble in hydrochloric acid, and which, therefore, are precipitated by sulphuretted hydrogen in presence of that acid. They are Mercury (ic), Lead, Bismuth, Copper, Cadmium, Tin, Antimony, and Arsenic.

Group III. includes *those of the remaining metals* whose hydrates are insoluble in water and in ammonium chloride solution, and which, therefore, are precipitated by ammonia in presence of ammonium chloride. They are Iron, Chromium, and Aluminium.

---

* These divisions and the groop reagents must be perfectly familiarised before commencing systematic analysis.

Group IV. consists of *those of the remaining metals* whose sulphides are insoluble in water, and which, therefore, are precipitated by ammonium sulphide in alkaline solutions. They are Nickel, Cobalt, Manganese, and Zinc.

Group V. includes, of *the remaining metals*, those whose carbonates are insoluble in water and ammonium salts, and which, therefore, are thrown down by ammonium carbonate in presence of ammonium chloride. They are Barium, Strontium, and Calcium.

Group VI. comprises *the remaining metals* for which there is no general reagent. They are Magnesium, Potassium, Sodium, and Ammonium.

Special notice should be taken of the words "of the remaining metals," which appear so often in the above account. Group IV., for example, does not consist of all the metals which are precipitated by ammonium sulphide in alkaline solutions, but only of those remaining after the removal of Groups I., II., and III., which are thus precipitated.

After determining the group to which a metal belongs, the next thing is to find which of the several metals in that group it is. Advantage is taken of the slight differences in the properties of the similar compounds of different metals existing in the same group—*e.g.*, as to colour, solubility in various reagents, &c.—in order to settle this question; and, finally, the answer is confirmed and substantiated by the application of a *special* or *confirmatory* test. In testing for the acid radicles the splitting up of the various acids into groups cannot be so fully carried out, and more importance is to be attached to confirmatory or special reactions.

It must be carefully borne in mind that analysis is founded upon a knowledge of the properties of the metals and acids, and is really an application of such chemical knowledge to the question in hand. The student should, therefore, in all cases endeavour to clearly understand the chemical changes which are produced in the various operations of chemical analysis. To this end he will derive great advantage by making it a rule to express by means of equations the various reactions which he performs. These equations should be written in a note-book at the time the experiments are being made in the laboratory.

It will be found that almost all reactions made use of in analysis are examples of metathesis or double decomposition, *e.g.*, the interchange of acids and bases—thus barium chloride and sodium sulphate give us sodium chloride and barium sulphate, the latter, being insoluble in water, is thrown down as a white precipitate.

$$Na_2SO_4 + BaCl_2 = BaSO_4 + 2NaCl.$$

Such reactions, of course, present little difficulty, but the practice of writing the equations should be adhered to, even in simple cases, as by this means the student acquires a valuable familiarity with the valency of metals and the basicity of acids.

ANALYSIS OF A SIMPLE SALT.

CHAPTER I.

INTRODUCTORY EXPERIMENTS.

To bend a piece of glass tubing.—Take a piece of $\frac{3}{16}$-inch soft tubing, and hold it horizontally in the upper part of an ordinary luminous gas flame, with its length in the plane of the flame. Turn the tubing slowly round and round so as to heat it uniformly. When it begins to soften, remove it from the flame and bend it slowly and carefully to the required shape. Allow to cool, and *then* remove the coating of soot from the bend.

To cut narrow glass tubing with a file.—Take a piece of glass tubing and lay it on a table. Taking hold of it with the finger and thumb of the left hand near the point where it is to be cut, draw one edge of a sharp triangular file *once* across it, from heel to point. The tube can now be broken by forcibly attempting to bend it away from the file-scratch, and at the same time trying to pull it apart. The cut ends must then have their sharp edges rounded by holding the tube vertically in the Bunsen flame, until the flame is coloured strongly yellow by the sodium of the glass.

To draw out a piece of glass tubing; to make a jet.—Take a short piece of tubing and heat the middle portion in a small blowpipe flame, supporting each end of the tube with the fingers, and continually turning it round so as to equally heat all sides; when the tube is quite soft, remove it from the flame and slowly draw it out. The more slowly it is drawn out, the more gradually will the tube taper towards the centre. Then with a file cut the tube at a suitable point, round off the cut ends in the flame, and the jet is ready for use.

To make a bulb tube.—Take a piece of $\frac{3}{16}$-inch tubing, about 6 inches long, and heat one end in the blowpipe flame until it is completely closed and rounded off. Before this rounded end becomes too thick, take it out of the flame, and, holding the tube vertical, blow into it steadily, but not too strongly, until a small bulb is blown at the heated end.

Use of Spatula.—It is often necessary to take a small quantity of some solid substance out of a bottle. For this purpose the spatula—an instrument very like a small table-knife—is used. A small bone or ivory paper-cutter answers the purpose very well. The blade of a penknife is sometimes used, but the use of iron or steel is objectionable. A platinum spatula is the best to use.

To fix a platinum wire in a glass holder.—Take 6 inches of narrow glass tubing and draw it out a little in the middle. Cut in two at the middle of the drawn-out part. Into the narrow cut end insert a piece of platinum wire about 2 inches long; then hold this end in the flame until the glass closes round the wire. A piece of glass rod makes a better holder. Draw the rod out and cut in two as described above. Then hold the narrow end of the rod in the flame until it is quite soft, and insert the piece of platinum wire into the soft end. The free end of the wire should be bent into a loop or coiled into a small flat spiral.

The platinum wire thus mounted will be in frequent use for flame tests, and should be kept dipping in hydrochloric acid in a test tube. This can be done by fitting the holder with a cork which fits into the mouth of the test tube. Kept in this way the wire is always clean.

Flame tests.—(1) Place a small quantity of solid sodium carbonate, Na_2CO_3, on a watch-glass. Lift a small portion of it with the loop of the platinum wire, and hold in the lower outer part of the Bunsen flame. The flame shows an intense yellow colour characteristic of sodium compounds. Clean the wire by holding it in the flame until all trace of the yellow coloration disappears. Then wash it in dilute HCl and again heat it.

(2) Take some barium hydrate (or any barium salt) and heat it in the same way. The flame is of a yellowish green colour, but possibly not very intense. Pour a few drops of HCl into a watch-glass, dip the loop of the wire in it, and take up with the moistened wire a few particles of the salt. On now placing the wire in the flame the yellowish green coloration will be much more intense. Now clean the wire by alternately dipping it in HCl and heating it in the flame. For a time the effect of this is merely to increase the intensity of the coloration, but after some time it becomes fainter, and by using fresh acid once or twice the wire can be thoroughly cleaned. Barium compounds are the most difficult to remove from the wire.

(3) Take some strontium nitrate and treat in the way described above. The flame is coloured an *intense* crimson. Moistening with HCl makes the flame more intense. Clean the wire, wash it and put it away in a corked test tube or bottle.

Borax bead reactions.—Borax, being an acid borate, $Na_2B_4O_7 = 2NaBO_2 . B_2O_3$, has the power of displacing other (volatile) acids from metallic salts, so forming fusible borates. These borates, in the case of many metals, possess distinctive colours, and so afford valuable tests.

(1) Take a piece of platinum wire fitted with a glass handle, as already described, but without a loop at the end, heat in the Bunsen flame, and dip into powdered borax. Some of the salt will adhere to the wire. Again heat the wire, when the borax upon it will first "intumesce," *i.e.*, swell up from the escape of its water of crystallization, and then fuse quietly to a clear, colourless bead. Now take a small quantity of a manganese com-

pound *e.g.*, $MnSO_4$ or MnO_2, and bring it into contact with the bead, and again heat in the *outer* part of the Bunsen flame, a characteristic *purple* or *amethyst* colour will be produced. If now the air-holes at the base of the lamp be partially closed, so as to produce a slight luminous appearance in the upper central portion of the flame, and the bead be heated in this flame ("reducing flame"), since there is an excess of coal-gas there, the oxide of manganese, existing as borate, in the bead will be reduced to a lower state of oxidation, and the bead will, when cooled (best in the coal-gas at the centre of the base of the Bunsen flame), be found to be colourless. Treated in a similar way, Chromium compounds give a *green* bead in either flame; Iron, a *reddish yellow* in the oxidizing, *greenish yellow* in the reducing flame; Cobalt, a *bright blue* in either flame, &c., &c. In all cases only a very small quantity of the substance to be tested must be used, otherwise the colour produced is too dark to be recognisable.

The Blowpipe.—To acquire the proper use of the blowpipe will necessitate a good deal of practice. A *continuous* blast of air must be kept up; for this purpose it is necessary to blow without interfering with the breathing. The secret of doing so is to use the cheeks to blow with, and to breathe through the nose. If the cheeks be blown out, it will be found that air can be forced out of the mouth by contraction of the cheeks at the same time as inspiration is effected through the nose. By continued practice this may be done continuously. When blowing continuously, however, since the air is forced out of the mouth by the pressure of the cheeks—not by their contraction—they must be kept distended. It is not necessary to blow hard: a very gentle blast suffices.

To obtain the blowpipe flame, the air-holes at the bottom of a Bunsen burner are closed so as to give a smoky luminous flame, and the blast of air from the nozzle of the blowpipe is then directed across this flame so as to produce a long straight pointed flame. It is used for observing the effects of a very high temperature on different substances, and in some cases to effect their oxidation or reduction.

To oxidize a substance it is heated in the outer flame—that is, at the tip of the blowpipe flame. To effect reduction, the inner flame must be used—that is, the substance is heated in the centre of the blowpipe flame, which for this purpose must show a small luminous streak in the centre.

Experiments with the blowpipe.—(1) Take a piece of hard charcoal and scrape a small groove near one end. In this groove place a small quantity of litharge (lead oxide) and direct on it a reducing flame. In a short time it will melt, and if the heating be continued long enough a globule of lead will remain on the charcoal. This is an example of reduction effected by the reducing-flame and the charcoal.

(2) Take some potassium nitrate, KNO_3, and, having placed it on charcoal as described in (1), bring the blowpipe flame to bear on it. It suddenly flares up, and burns a portion of the charcoal with it. This is *deflagration*.

(3) Take weights of powdered sodium carbonate, Na_2CO_3, and potassium carbonate, K_2CO_3, proportionate to their molecular weights, and mix them thoroughly. This mixture is known as *fusion mixture*, and should be kept ready for use. Take a small quantity of litharge (lead oxide), mix it with three or four times its weight of *fusion mixture*, and heat the mixture on charcoal before the blowpipe. The mass fuses, and in a short time small globules of lead appear and are readily fused into one large globule. The use of the fusion mixture is twofold—it prevents the fused litharge from being absorbed by the charcoal, and the fusion and reduction are more easily effected than in experiment (1). The fusion mixture here acts as a *flux*.

Note.—When a substance is to be *reduced*, it should be heated *on charcoal*, with some suitable flux, in the *reducing flame*. If *oxidation* is to be effected, it should be heated on the platinum wire, or on a piece of platinum foil in the oxidising flame.

(4) Take some zinc oxide, ZnO, mix it carefully with fusion mixture, and heat on charcoal before the blowpipe. The oxide is reduced to the metallic state, and zinc, being volatile, burns on passing through the outer flame with a bluish green flame, forming the oxide which is deposited on the charcoal in the form of an *incrustation*, which is yellow when hot and white when cold. Now moisten the mass with a few drops of cobaltous nitrate, and again heat in the outer flame. The mass now assumes a fine green colour.

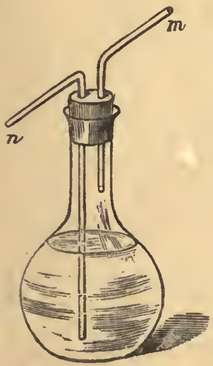
(5) Take a small quantity of tin oxide, and mix it with powdered potassium cyanide, KCy; place the mixture on charcoal, and cover it over with a little KCy, then heat before reducing blowpipe flame. The tin oxide is quickly reduced, and small globules of metallic tin are obtained. Substances that are not easily reduced by charcoal alone are readily reduced in the presence of KCy, owing to the tendency of that compound to take up oxygen or sulphur to form KCNO or KCNS.

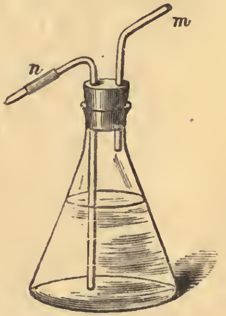
(6) Powder some copper sulphate, and mix it with five or six times its weight of fusion mixture. Heat the mixture on charcoal before the inner blowpipe flame. The mass fuses, and the $CuSO_4$ is reduced to metallic copper, which is too infusible to be fused into a globule, and therefore remains on the charcoal as small reddish particles or scales which are not easily detected. Allow the mass to cool, remove it from the charcoal together with the crust of underlying charcoal, and place the whole in a small mortar. Powder it very finely, and transfer the powder to a small beaker. Pour on some water, and stir the whole for some time; on standing, the metallic particles sink quickly, so that if the water be poured off after a short time the copper will be found as a powder at the bottom of the beaker, and may be recognised by the effect of adding a few drops of nitric acid. Iron compounds may be similarly dealt with, and the iron particles are readily detected by means of a magnet.

Sublimation.—(1) Take some ammonium carbonate, and place a small quantity in the bulb of a bulb-tube. Holding the stem of the tube with a pair of crucible tongs, heat the substance in the Bunsen flame. It gradually disappears from the bulb and condenses as a *white sublimate* in the upper part of the tube.

(2) Take some white arsenic, As_2O_3, and heat in a bulb-tube. A glittering white sublimate of As_2O_3 in octahedral crystals is obtained in the upper part of the tube. Mix some As_2O_3 with KCy and powdered charcoal, and heat the mixture in a bulb-tube. A brilliant black metallic mirror of arsenic is obtained in the upper part of the tube.

To make a Wash-Bottle.—Take a 16-oz. flask, either of the ordinary round form or, better, of the conical shape, fit it with a good cork or indiarubber stopper, in which bore two holes, in the first case by means of a round file, in the case of the rubber cork by a sharp cork-borer moistened with alcohol. Then fit two pieces of tubing bent as shown in the figure, taking care to round off the ends in the flame, and that the tube *n* ends very close to the bottom of the flask; to the end *n* of the longer tube fit a piece (about $1\frac{1}{2}$ inches) of narrow ($\frac{1}{8}$ in. diam.) indiarubber tubing, carrying at the other end a short piece





(about 1 inch) of glass tubing drawn out to a jet. The wash-bottle is to be nearly filled with distilled water, which can be expelled by blowing down the tube *m*, and the fine stream issuing from the jet can be directed in any desired direction by the flexibility of the rubber tubing. The wash-bottle is used chiefly for washing precipitates on filters and as a reservoir of pure water for general purposes.

To test the solubility of substances.—(1) Take some Epsom salts, $MgSO_4$, and with a spatula place a very small quantity of it in a dry test tube. Half fill the test tube with water from the wash-bottle, using the jet of water to wash all traces of the salt down the sides of the tube. Close the tube with the thumb, and shake up the solution. It will then be found that all the substance has dissolved.

(2) Take a small quantity of lead chloride, $PbCl_2$, and attempt to make a solution of it in the way described above. It will be found that this salt does not dissolve readily in cold water. Now hold the test tube in the Bunsen flame so as to boil the water. The lead chloride soon dissolves, and a clear solution is obtained. Allow this solution to stand and cool—the lead chloride again crystallizes out.

(3) Take some barium hydrate and attempt to make a solution as above. It will be found that this substance is apparently insoluble in both hot and cold water. Take some red litmus paper and test the solution, it will be found to give a strong alkaline reaction, thus showing that a solution has been obtained, and that barium hydrate is slightly soluble in water. Also, place a few drops of the solution on a piece of clean platinum foil and heat over the Bunsen flame. A small white speck is left on the foil showing that the solution contained some solid substance.

(4) Take some pure barium carbonate and treat as above. It will be found insoluble in water (unless, as is frequently the case, it contains traces of the hydrate.) Take a fresh quantity of the substance in a test tube and pour in some dilute hydrochloric acid, HCl. The car-

bonate rapidly dissolves with *effervescence*, carbonic acid gas, CO_2, being evolved.

Precipitation is the basis of testing in the wet way. One of Berthollet's laws of double decomposition may be enunciated thus:—

"Two substances in solution will always decompose each other if it be possible, by double decomposition, to produce another substance less soluble than either of the original substances."

This law expresses the principle of testing by precipitation. For example, take a solution of barium chloride in a test tube and add to it a small quantity of a solution of magnesium sulphate, $MgSO_4$. The following double decomposition is possible:—

$$\mathbf{Ba}Cl_2 + Mg\mathbf{SO_4} = \mathbf{BaSO_4} + \underset{\text{(soluble)}}{MgCl_2}.$$

Now barium sulphate, $BaSO_4$, is quite insoluble in water and acids, and therefore the reaction takes place and a white precipitate of $BaSO_4$ is produced.

This reaction now furnishes us with a test for the detection of barium compounds and also for sulphates; for the test is based on the insolubility of $BaSO_4$, and may therefore indicate Ba or H_2SO_4, but has nothing to do with Mg or HCl. A consideration of the reaction given below will further explain this:—

$$\mathbf{Ba}\,(NO_3)_2 + Na_2\mathbf{SO_4} = \mathbf{BaSO_4} + \underset{\text{(soluble)}}{2NaNO_3}.$$

Substances employed in systematic analysis for the detection of other substances are called *reagents*.

Solutions of gases or of solids, with other liquids so employed, are called "*wet*" reagents; those used in the solid form, "*dry*" reagents. Solutions used as wet reagents should not be too concentrated.

Filtration.—*To fold a filter paper*—If, as is usually the case, the precipitate is required as well as the clear liquid, an ordinary plain filter answers best. In cases where the amount of precipitate is only small, and the filtrate is the portion desired, a ribbed or folded filter should be used, as the liquid flows through such a paper much more quickly than through a plain filter.

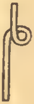
To fold a plain filter.—Take a circular piece of filtering paper of the requisite size, and fold it in half. Then again fold it in half, so as to form a quadrant. Now open it in such a way that a cone of paper is formed having one thickness of paper on one side and three thicknesses on the other. Take a plain funnel (*i.e.*, not a fluted one) having an angle of 60° at the apex, and fit the filter paper carefully into it, and moisten the paper with water. The paper should be in contact with the glass in every part, and ought in all cases to be small enough to go entirely into the funnel, and leave a small portion of the glass uncovered all round its upper edge.

If, as sometimes happens, the angle of the funnel is not exactly 60°, it will be found that the paper will not fit accurately to the glass. In this case, fold the filter paper the second time not exactly in half, and open the smaller or larger side according to whether the angle of the funnel is less or greater than 60°.

A little care in folding and fitting the paper to the funnel is well repaid by the greater rapidity with which the liquid will flow through; and, since filtration is the most tedious operation in analytical work, any means by by which it can be facilitated is well worth attention.

Certain precipitates—*e.g.*, silver chloride—filter easily and rapidly in the usual way, whilst others—*e.g.*, aluminium or ferric hydrate—filter very slowly by the ordinary method. In order to increase the rapidity of filtration in such cases, the following arrangement may be used:—

The funnel and filtering paper are arranged as already described, but, instead of allowing the filtrate to drop directly from the funnel into the test tube or other vessel, fit to the lower end of the funnel, by means of a short length of indiarubber tubing, a piece of narrow glass tube bent round upon itself, near its upper extremity, as in the diagram:—



The bend insures that the liquid shall entirely fill the tube, and there is therefore formed an unbroken column of liquid in the lower part of the tube, and this column by its weight, reduces the pressure on the lower side of the filter paper, and hence very greatly increases the rate of filtration. In using this arrangement it is important that the paper fits closely to the funnel, otherwise channels are formed for the admission of air, which, of course, altogether spoils the action of the long tube in reducing the pressure below the paper. The tube should be of as great a length as the filter-stand will allow of, but a length of a few inches has a remarkable effect in accelerating filtration, especially of gelatinous precipitates like ferric hydrate, a precipitate which is most tedious to filter by ordinary means. In making the bend in the tube, it will be found best to heat the tube at the proper place in a large flat flame, and, holding each end of the tube in the fingers, when the portion in the flame becomes quite soft, to bring the hands near together, at the same time giving one hand a slight twist. A loop will thus be formed, in a similar manner to the forming of a loop in a short piece of string, if it be held by one hand at each end, and the hands be then brought nearer together. A little practice will make this clearer than any amount of description.

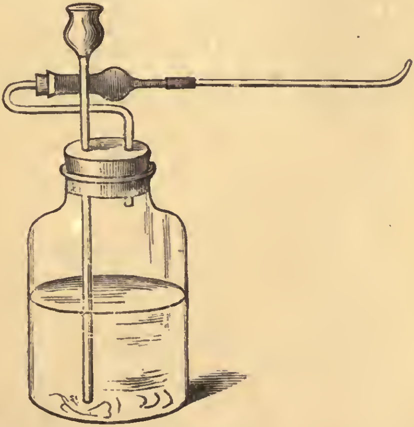
To fold a pleated or folded filter paper.— Take a circular piece of filtering paper and fold it in half, then again, and again in half, then open so that its outline is a semicircle; the creases produced by the other foldings are merely to serve as guides. Then, beginning at the straight side (*i.e.*, the diameter of the circle of which the paper now forms half), fold the paper from the centre to the circumference as far as the first crease; then turn the whole paper over, and fold the folded portion back an equal amount on the other side; and so on, until the whole paper is folded into a sector-shaped piece. Press the paper well so as to render the creases distinct, and then open it. A conical, pleated filter will thus be formed which, when supported by a funnel, will allow a liquid to flow through with great rapidity.

Filtration.—Take a solution of barium chloride, $BaCl_2$, and add a solution of sodium carbonate, Na_2CO_3, slowly, and finally drop by drop, until a pp. is no longer formed. Fit a filter-paper into a funnel, and moisten it with a jet of water from the wash-bottle to make it fit closely to the glass. Then support the funnel on a suitable stand, and, having placed a test tube or small beaker below to receive the *filtrate*, pour a portion of the contents of the test tube gently on to the side of the filter-paper, taking care not to fill it too full. When this portion has filtered through, add another portion, and so on until the whole is filtered. To *wash* the pp. on the filter-paper, direct a jet of water on to it so as to wash it down into the apex from the upper edge of the paper, fill up with water, and allow to stand until this water has all filtered through. Then fill up again several times until the washings show no alkaline reaction.

Testing solubility of precipitates.—Take a small quantity of a solution of silver nitrate, $AgNO_3$, dilute it with water, and add dilute hydrochloric acid, HCl; a white curdy pp. of silver chloride, AgCl, is obtained. Add a solution of ammonia, AmHO, in excess, and shake up the mixture: the pp. dissolves, showing that AgCl is soluble in AmHO.

This is a convenient method of procedure applicable in most ordinary cases, but it is sometimes advisable to separate the pp. from the accompanying liquid. This can be done thoroughly by filtration and washing, but more readily thus:—Allow the pp. to subside, pour off the liquid and fill up with water. Again allow the pp. to settle down and pour off the water. Repeat this once or twice. This method of washing by *decantation* is applicable only to heavy precipitates.

Marsh's Apparatus.—Take a wide-mouthed bottle of about 8 ounces capacity, fit it with a good cork or india-rubber stopper, in which bore two holes, one for an acid funnel, the other for a delivery tube; this should be bent back horizontally for 3 inches, and then back upon itself and cut off short; then fit it by means of a small cork to a small calcium chloride tube in order to dry the hydrogen. Connect the other end of this tube with a straight piece of hard glass tubing turned up at the end and drawn out to a jet. It is quite common, however, to use an ordinary form of a delivery tube drawn out to a jet. Place zinc (free from arsenic*) in the bottle and pour upon it pure sulphuric acid diluted with about five times its volume of water. Allow the generation of hydrogen to proceed for a few minutes in order to expel all air from the apparatus, and then light the jet. If the materials are pure, the flame will be almost colourless, and will yield no stain if a piece of porcelain be held in the flame (see p. 40). Great care must be taken that the jet is not lighted too soon; otherwise serious consequences may result, a mixture of hydrogen and air being highly explosive.



Sulphuretted Hydrogen.—This is best used in the form of gas, though a solution in water will suffice if the apparatus for the manufacture of the gas be not readily available. The best form of apparatus consists of two bottles with necks in the side connected together by a piece of indiarubber tubing. One bottle is partially, about half, filled with a mixture of fragments of iron sulphide and glass marbles, the latter being used to allow of the free passage of a liquid in the interstices between the pieces of iron sulphide; the other bottle contains dilute hydrochloric acid (1 of strong acid to about 12 or 15 of water). The upper neck of the bottle containing the iron sulphide is fitted with a cork carrying a tube which is bent at right angles and fitted either with a piece of indiarubber tubing and a pinch-cock, or, better, with a glass stop-cock. When in use, the acid bottle is raised on a block and a piece of straight glass tube is fitted, by means of a short length of indiarubber tubing, to the delivery tube of the other bottle, and the flow of the gas governed by the stop-cock or pinch-cock. When not in use, the position of the bottles is reversed, when the acid runs away from the iron sulphide, and all action ceases. This arrangement is shown in the figure.

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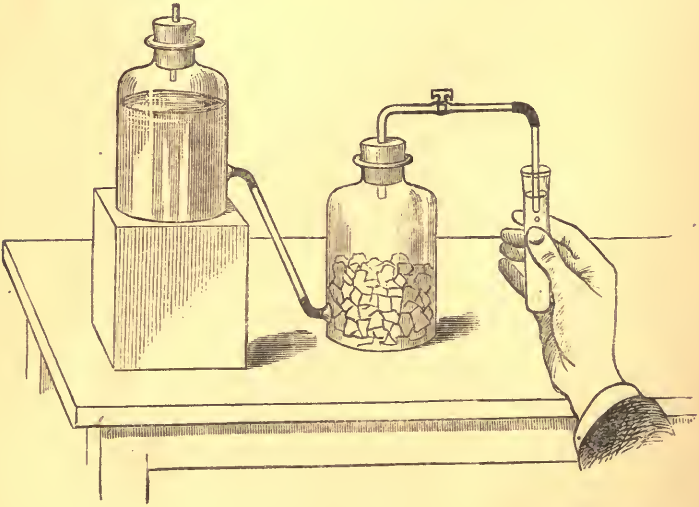
* Commercial zinc contains arsenic; it would not therefore afford reliable evidence in testing matter for arsenic (*e.g.*, the contents of a stomach where arsenical poisoning has been suspected).



CHAPTER II.

REACTIONS OF THE METALS.

As mentioned in our Introduction, there are certain reagents which will precipitate any one of a group of metals, and hence, by a judicious choice of such reagents applied in the proper order, the metals may be divided into groups. The method of analysis adopted in the following pages divides the metals into six groups, the first five being each distinguished by a special group reagent.

By this division into groups, the work of analysis is much simplified; for, by successive application of the group tests, we readily determine the group to which the given substance belongs. In this way the field of investigation is narrowed, and it only remains to determine, by special tests, what particular member of the group we have to deal with. For this reason it is important to note in the following reactions the special tests which serve to distinguish the different members of each group from one another.

After going through all the tests for the members of each group, Table A on page 78 may be followed, until the group is determined, and then the corresponding Table which succeeds it worked through.

GROUP I.

METALS PRECIPITATED AS CHLORIDES.

Group-reagent HCl.

SILVER, Ag . . .	**Pp.**	**Silver Chloride, AgCl**	(*white*).
LEAD, Pb	,,	**Lead Chloride, $PbCl_2$**	,,
MERCURY, 'Hg' . . (*Mercurous*)	,,	**Mercurous Chloride, Hg_2Cl_2**	,

SILVER, **Ag.**

Solution—Silver Nitrate, $AgNO_3$.

1. The group-reagent, hydrochloric acid, HCl, gives a *white curdy* pp. of AgCl, which becomes violet on exposure to the light: *insoluble in hot water and nitric acid, soluble in ammonia.*

2. Potassium chromate, K_2CrO_4, gives, in *neutral*[*] solutions, a *dark red* pp., which, on the addition of HCl, becomes white on account of the formation of silver chloride.

3. Potassium cyanide, KCy, gives a *white curdy* pp., very soluble in excess of KCy. Rubbed upon a bright piece of copper, a white stain of silver is produced. This stain does not disappear on heating.

4. On mixing with sodium carbonate, Na_2CO_3, and heating on charcoal before the blowpipe, brilliant white malleable beads of silver are obtained.

Silver nitrate is the only common soluble salt of silver.

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[*] Solutions are sometimes given containing free acid. In that case tests of this kind cannot be relied on. (See p. 77.)

LEAD, **Pb.**

Solution—Lead Nitrate, $Pb(NO_3)_2$.

1. Group-reagent, HCl, gives a *heavy white crystalline* pp. of lead chloride, $PbCl_2$: *soluble in hot water*, but crystallizing out on cooling; *insoluble in ammonia.* No pp. is formed in dilute solutions.

2. Potassium chromate, K_2CrO_4, gives a *bright yellow* pp. of lead chromate, $PbCrO_4$: insoluble in dilute nitric acid and acetic acid; soluble in sodium hydrate, NaHO. [*Distinction from Bismuth.*]

3. Potassium iodide, KI, gives a *golden yellow* pp. of lead iodide, PbI_2: soluble in boiling water, but crystallizing out in brilliant iridescent scales on cooling.

4. Dilute sulphuric acid, H_2SO_4, gives at once in concentrated solutions a white pp. of lead sulphate, $PbSO_4$: precipitation takes place in very dilute solutions only after a time; the addition of alcohol favours the precipitation.

5. Heat on charcoal with sodium carbonate, Na_2CO_3, and potassium cyanide, KCy: beads of lead are formed, which are malleable, and mark paper.

Common soluble salts. Lead nitrate, $Pb(NO_3)_2$, and lead acetate, $Pb(C_2H_3O_2)_2$, (sugar of lead), are readily soluble in cold water. The latter, being a salt of an organic acid, belongs strictly to Organic Chemistry. Lead chloride, $PbCl_2$, and lead iodide, PbI_2, are sparingly soluble in cold water, but readily soluble in hot water.

MERCURY (*mercurous*), **'Hg'**.

Solution—Mercurous Nitrate, $Hg_2(NO_3)_2$.

1. Group-reagent, HCl, gives a *white* pp. of mercurous chloride, Hg_2Cl_2 (*calomel*): *insoluble in hot water, blackened by ammonia, AmHO.* The blackening on addition of AmHO is due to the formation of $NH_2'Hg'_2Cl$, mercurous-ammonium chloride.

2. Sodium hydrate, NaHO, gives a *black* pp. of mercurous oxide, Hg_2O. [*Distinction from mercuric salts*, which give a *dark yellow* pp. of mercuric oxide, HgO.]

3. Stannous chloride, $SnCl_2$, gives a *grey* pp. of metallic mercury. [*Distinction from mercuric salts*, see p. 35.]

4. When any dry mercurous compound is heated in a tube with powdered charcoal and Na_2CO_3, the metal is reduced and volatilized, and deposited in the cooler parts of the tube in the form of a mirror made up of minute metallic globules.

5.* Acidulate a portion of a solution of any mercurous compound in a tube with hydrochloric acid, and boil with a strip of pure electrolytic copper. A film of mercury is deposited on the copper. When dried between blotting-paper and heated in a tube, the mercury sublimes and the copper resumes its natural colour.

Mercurous nitrate, $Hg_2(NO_3)_2$, is the only common soluble mercurous salt.

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* Compare Reinsch's test for Arsenic (p. 41).

GROUP II.

METALS PRECIPITATED AS SULPHIDES.

Group-reagent—H_2S in presence of HCl.

DIVISION I.

Metals whose Sulphides are Insoluble in Alkaline Sulphides or Hydrates.

MERCURY, Hg'' (*mercuric*).	**Pp.**	**Mercuric Sulphide, HgS**	(*black*).
LEAD,* Pb	,,	**Lead Sulphide, PbS**	,,
BISMUTH, Bi	,,	**Bismuthous Sulphide, Bi_2S_3**	,,
COPPER, Cu	,,	**Cupric Sulphide, CuS**	,,
CADMIUM, Cd	,,	**Cadmium Sulphide, CdS**	(*yellow*).

DIVISION II.

Metals whose Sulphides are Soluble in Alkaline Sulphides or Hydrates.

TIN, Sn'' (*Stannous*)	**Pp.**	**Stannous Sulphide, SnS**	(*dk.brown*).
Sn^{iv} (*Stannic*)	,,	**Stannic Sulphide, SnS_2**	(*yellow*).
ANTIMONY, Sb	,,	**Antimony Sulphide, Sb_2S_3**	(*orange*).
ARSENIC, As	,,	**Arsenic Sulphide, As_2S_3**	(*yellow*).

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* Lead, as $PbCl_2$, is slightly soluble in cold water, and therefore occurs in Group II. as well as in Group I.

The sulphides dissolve in ammonium sulphide or caustic soda because of the formation of sulpho-salts.

Thus in the case of As_2S_3,

$$As_2S_3 + 3(NH_4)_2S = 2(NH_4)_3AsS_3.$$

The compound formed, which is soluble in water, is the analogue of ammonium arsenite, $(NH_4)_3AsO_3$, sulphur replacing oxygen, and is hence called *ammonium sulpharsenite.* With NaOH the reaction is—

$$As_2S_3 + 6NaOH = Na_3AsS_3 + Na_3AsO_3 + 3H_2O.$$

In the case of antimony sulphide, similar reactions occur; but in the case of tin a slight difference is found. With stannic sulphide the reactions are—

$$SnS_2 + (NH_4)_2S = (NH_4)_2SnS_3,$$ *Am. sulphostannate,*

and $$2SnS_2 + 4NaOH = Na_2SnS_3 + Na_2SnO_3 + H_2S + H_2O;$$

but with stannous sulphide, *colourless* ammonium sulphide will not produce a soluble compound; *yellow* ammonium sulphide, however, since it owes its colour to persulphides, first converts the SnS into SnS_2, and then dissolves it as above. Caustic soda will usually dissolve SnS because of the sulphur with which the latter is usually mixed, and forms, as before, the sulphostannate and stannate.

DIVISION I.

MERCURY, **Hg″**.

Solution—Mercuric Chloride,* $HgCl_2$.

1. The group-reagent,* sulphuretted hydrogen, H_2S, added slowly, gives first a *white* pp., which, as more of the reagent is added, first becomes *yellow*, then *brown*, and finally *black*. The black pp. is HgS.

2. Stannous chloride, $SnCl_2$, gives, with mercuric salts, a *white* pp. of mercurous chloride, Hg_2Cl_2, which, on boiling with excess of the reagent, gives grey metallic mercury. If the solution be poured off, and the pp. boiled with hydrochloric acid, the mercury is obtained in small globules.

3. Potassium iodide, KI, gives a pp. which is first yellow, but changes to a *bright scarlet* pp. of mercuric iodide, HgI_2; this pp. is soluble in excess of the mercuric salt or of potassium iodide.

4. Sodic hydrate, NaHO, when added in excess, gives a *yellow* pp. of mercuric oxide, HgO: insoluble in excess.

5. Repeat reactions 4 and 5 for Hg′. Similar results will be obtained.

Mercuric chloride, $HgCl_2$ (corrosive sublimate) is the most important soluble mercuric salt. Mercuric nitrate, Hg $(NO_3)_2$, is also soluble, but the salt is unstable, and when in solution tends to form insoluble basic salts.

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* Sulphuretted hydrogen may be used as a solution in water, or the gas itself may be passed into the solution from a suitable apparatus (see p. 28) until the precipitation is complete. The latter method, where practicable, is preferable.

LEAD, **Pb.**

Unless the solution be very dilute, lead is usually detected in Group I. If not so detected, a black pp. of lead sulphide, PbS, will be obtained on addition of H_2S in Group II., if the solution be not too acid. For this reason, if there is a doubtful indication of a pp. in Group I., and lead is suspected from preliminary examination (see p. 74), H_2S should be passed through the original solution if that be acid, and not through the portion to which HCl has been added.

[This point is of importance in systematic analysis. On account of the slight solubility of $PbCl_2$ in cold water, Pb is not completely precipitated in Group I.; and if the solution be too acid, or too concentrated, it is not precipitated at all in Group II., and therefore appears in Group III., where it is often mistaken for some other metal.]

For other reactions of Lead, see Group I.

BISMUTH, **Bi.**

Solution—Bismuth Nitrate, $Bi(NO_3)_3$, dissolved in dilute nitric acid.

1. H_2S gives a *brownish-black* pp. of bismuthous sulphide, Bi_2S_3.

2. Water added in large quantity to a solution of bismuth, to which HCl or ammonium chloride, AmCl, has first been added, **produces a white pp.** of a basic chloride BiOCl, which is insoluble in tartaric acid. [*Distinction from the basic chloride of antimony, SbOCl*, which is soluble in tartaric acid.]

3. Potassium chromate, K_2CrO_4, gives a *yellow* pp. of the basic chromate, $Bi_2(CrO_4)_2O$, soluble in dilute nitric acid, but insoluble in sodium hydrate. [*Distinction from lead.*]

4. Dilute sulphuric acid, H_2SO_4, gives no pp. [*Distinction from lead.*]

5. Mix with sodium carbonate, Na_2CO_3, and heat on charcoal before the blowpipe in the reducing flame: brittle metallic beads are obtained, and an *orange yellow incrustation* is formed on the charcoal.

Most of the soluble bismuth salts are decomposed by water, insoluble basic salts being formed. Bismuth nitrate, $Bi(NO_3)_3$, the commonest salt dissolves in a small quantity of water, especially if nitric acid is present. A large quantity of water decomposes the salt, and basic bismuth nitrate is formed.

COPPER, **Cu.**

Solution—Cupric Sulphate, $CuSO_4$.

1. H_2S gives a *brownish-black* pp. of cupric sulphide, CuS: insoluble in hot dilute sulphuric acid.

2. Ammonia, AmHO, or ammonium carbonate, Am_2CO_3, when added in small quantities, produces a *greenish-blue* pp. of a basic salt, which is readily dissolved in excess of the reagent, forming a splendid *azure-blue liquid;* this coloration is easily perceptible, even if the solution contain only slight traces of copper. The blue solution contains ammonio-cupric sulphate, $(NH_3)_2Cu''SO_4$.

3. Potassium ferrocyanide, K_4FeCy_6, gives a *reddish-brown* pp. of cupric ferrocyanide, Cu_2FeCy_6, in neutral or slightly acid solutions, but not in alkaline solutions. A *brownish colour* is produced in very dilute solutions of copper salts; it is best seen when the reaction takes place in a watch-glass placed on a sheet of white paper. K_4FeCy_6 supplies one of the most delicate reactions for copper salts.

4. Potassium cyanide, KCy, gives a *greenish-yellow* pp. of cupric cyanide, $CuCy_2$, which is soluble in excess. H_2S produces no pp. from this solution.

5. Sodium hydrate, NaHO, gives a *bulky light blue* pp. of cupric hydrate, $Cu(HO)_2$; if excess of NaHO be added, and the mixture boiled, the pp. becomes black, having been converted into cupric oxide, CuO.

6. If a piece of bright iron be dipped into a cupric solution acidulated with HCl, it becomes coated with copper; if clean iron filings be boiled in the solution, it will lose its blue colour, the copper being wholly displaced by the iron.

7. Mix with sodium carbonate, Na_2CO_3, and potassium cyanide, KCy, and heat before the inner blowpipe flame for some time: red metallic scales of copper are obtained.

Cupric sulphate, $CuSO_4$ (blue vitriol) is the commonest soluble salt of copper. Cupric nitrate, $Cu(NO_3)_2$ and cupric chloride, $CuCl_2$, are also readily soluble.

CADMIUM, **Cd.**

Solution.—Cadmium Chloride, $CdCl_2$.

1. H_2S gives a *fine yellow* pp. of cadmic sulphide, CdS, *soluble in dilute sulphuric acid;* the precipitation is prevented by presence of much HCl. [*Distinction from copper.*]

2. Sodium hydrate, NaHO, gives a *white* pp. of cadmic hydrate, $Cd(HO)_2$: insoluble in excess.

3. Potassium cyanide, KCy, gives a *white* pp. of cadmic cyanide, $CdCy_2$: soluble in excess. H_2S precipitates from this solution CdS. [*Distinction from copper.*]

4. Mix with sodium carbonate, Na_2CO_3, and heat on charcoal before the inner blowpipe flame: a *reddish yellow incrustation* of cadmium oxide, CdO, is obtained. (*This experiment must be carefully performed, for if heated too strongly the oxide is completely volatilized.*)

Cadmium chloride, $CdCl_2$, cadmium sulphate, $CdSO_4$, and cadmium nitrate, $Cd(NO_3)_2$ are common easily soluble salts of cadmium.

DIVISION II.

TIN (*Stannous*), **Sn″**.

Solution—Stannous Chloride, $SnCl_2$.

1. H_2S gives a *chocolate-coloured* pp. of stannous sulphide, SnS: insoluble in ammonic carbonate, Am_2CO_3.

2. Mercuric chloride, $HgCl_2$, produces first a *white* pp. of Hg_2Cl_2, which, when boiled with excess of the solution, becomes of a *greyish-black colour* owing to its reduction to metallic mercury.

3. Auric chloride, $AuCl_3$, gives a purple pp. of doubtful composition (*purple of Cassius*). The probable reaction is

$$2AuCl_3 + 3SnCl_2 + 6H_2O = \underbrace{Au_2 + 3SnO_2}_{\text{pp.}} + 12HCl.$$

4. Add a strip of zinc to a solution acidulated with HCl: it becomes coated with a spongy mass of metallic tin. Platinum-foil, in contact with the strip of zinc, does not becomes blackened. [*Distinction from copper and antimony.*]

5. Mix with sodium carbonate, Na_2CO_3, and potassium cyanide, KCy, and heat on charcoal before the blowpipe: malleable beads of tin and a white incrustation on charcoal are obtained.

TIN (*Stannic*), **Snⁱᵛ**.

Solution—Stannic Chloride, $SnCl_4$.

1. Sulphuretted hydrogen, H_2S, gives a *dull yellow* pp. of stannic sulphide, SnS_2: *insoluble in* Am_2CO_3.

2. Mercuric chloride, $HgCl_2$, gives no pp. [*Distinction from stannous salts.*]

3. Reactions 4 and 5 for stannous compounds apply also to stannic compounds.

$SnCl_2$ and $SnCl_4$ are the only common soluble salts of tin.

ARSENIC,* **As.**

Solution—Arsenious Oxide, As_4O_6, dissolved in dilute HCl.

1. Sulphuretted hydrogen, H_2S, gives (*in acid solutions*) a *lemon yellow* pp. of arsenious sulphide, As_2S_3: *insoluble in strong boiling HCl; readily soluble in a concentrated solution of ammonium carbonate,* Am_2CO_3, boiling if necessary.

2. Add cupric sulphate, $CuSO_4$, and then sodium hydrate, NaHO, drop by drop, till in excess (to render the solution alkaline): a *fine green* pp. of arsenite of copper ($HCuAsO_3$) is obtained.

3. Marsh's Test.—Hydrogen is generated from *pure* zinc and *pure* dilute sulphuric acid in the apparatus described on p. 26, and the jet lit as soon as all the air in the apparatus is displaced by hydrogen. A small quantity of the solution is then poured down the acid funnel into the bottle. In a short time arseniuretted hydrogen, AsH_3, is produced and escapes, mixed with a large proportion of hydrogen. The escaping gas now burns with a bluish flame, and fumes of As_4O_6 are evolved. If a cold porcelain dish be held in the flame, a bright black stain is produced on the dish. *This stain is readily dissolved by a drop of sodium hypochlorite, NaOCl.* [*Distinction from antimony.*]

If, while the gas is still escaping, the delivery tube be heated by the flame of a Bunsen burner or spirit lamp, at a point a short distance from the jet, a black ring will be produced on the tube just beyond the flame. If the tube be now cut off at this point, and held in a slanting position over a flame so that the ring is gently heated, a sublimate of octahedral crystals will be formed at the jet end of the tube. If the tube, with the sublimate, be dropped into a test tube containing dilute HCl, the As_4O_6 dissolves and a solution is obtained from which H_2S will precipitate As_2S_3 as in test 1.

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* Arsenic, when present as an arseniate, that is, as As^v, is only precipitated as As_2S_3 *by passing the solution through the gas for some time.* In this form arsenic will be detected as an *acid,* (see page 67).

4. **Hoffmann's Test.**—By the aid of the apparatus employed for the previous test, generate arseniuretted hydrogen, AsH_3; but, instead of burning the gas, pass it by means of a piece of bent tubing into a test-tube containing a solution of silver nitrate. A black pp. of metallic silver is produced, whilst nitric and arsenious acid remain in solution—

$$AsH_3 + 6AgNO_3 + 3H_2O = 3Ag_2 + H_3AsO_3 + 6HNO_3.$$

When the gas has been passing for some minutes, filter the liquid in the tube, add some more silver nitrate and then a few drops of solution of ammonia for the purpose of neutralizing the nitric acid. A *yellow* pp. of Ag_3AsO_3 is obtained.

5. **Fleitmann's Test.**—Boil a piece of granulated pure zinc or aluminium foil with a solution of caustic soda: hydrogen is given off. Add a few drops of a solution of As_4O_6; the escaping gas now contains AsH_3, which is recognised by its blackening a piece of filter paper previously moistened with $AgNO_3$, and held over the mouth of the test-tube. [*Distinction from antimony*, the compounds of which are not reduced in alkaline solutions to SbH_3.]

6. **Reinsch's Test.**—The solution is mixed with HCl, and boiled with a clean piece of copper-foil. A grey film is deposited on the copper. The copper-foil, having been washed and dried, is then heated in a narrow open tube where the arsenic oxidises, and arsenious anhydride, As_4O_6, is deposited in the cool part of the tube in octahedral crystals.

7. Mix with sodium carbonate, Na_2CO_3, and powdered charcoal, and heat in a small bulb-tube: a black *lustrous* metallic ring will be produced in the cool part of the tube. This ring, when treated as in Test 3, gives exactly the same results.

Arsenious oxide, As_4O_6, slightly soluble in water, but readily soluble in dilute HCl is the only common arsenic compound.

ANTIMONY, **Sb.**

Solution—Antimonious Chloride, $SbCl_3$, dissolved in dilute HCl.

1. Sulphuretted hydrogen, H_2S, gives (*in acid solutions*) an *orange-red* pp. of antimonious sulphide, Sb_2S_3: *insoluble in* Am_2CO_3.

2. Water produces a white milkiness due to pp. of antimony oxychloride, SbOCl: *easily soluble in tartaric acid.* [*Distinction from bismuth.*]

3. If a piece of platinum-foil and then a strip of zinc be dropped into a solution of antimony acidulated with HCl, so that the metals touch one another, a *brown* or *black* film (metallic antimony) will coat the platinum: free nitric acid must, however, not be present.

4. **Marsh's Test.**—Apply this test as described for arsenic. On the introduction of the antimony solution to the apparatus, antimoniuretted hydrogen is produced, and the flame becomes bluish green in colour. A black stain is produced by this flame on cold porcelain, just as in the case of arsenic; but the *stain is less lustrous, and is insoluble in sodium hypochlorite*, NaOCl.

5. **Hoffman's Test.**—Apply this test as for arsenic. A *black* pp. is obtained in the solution of silver nitrate: this contains antimony as $SbAg_3$, whilst nitric acid remains in solution—

$$SbH_3 + 3AgNO_3 = SbAg_3 + 3HNO_3.$$

6. Mix with sodium carbonate, Na_2CO_3, and potassium cyanide, KCy, and heat on charcoal before the inner blowpipe flame: brittle beads of antimony are produced which give off white fumes of antimonious oxide, Sb_2O_3.

Antimonious chloride, $SbCl_3$ (*butter of antimony*) is the commonest antimony compound. It readily dissolves in a small quantity of water or in dilute HCl. A large quantity of water precipitates SbOCl.

GROUP III.

METALS PRECIPITATED AS HYDRATES BY AmHO IN THE PRESENCE OF EXCESS OF AmCl.

Group-Reagent, AmCl and AmHO.*

IRON, Fe‴ (*Ferric*) . **Pp. Ferric Hydrate, $Fe_2(HO)_6$** (*reddish-brown*).

[IRON, Fe″ (Ferrous). Ferrous salts are not completely precipitated in the presence of AmCl; hence, before testing for iron in this group, a few drops of nitric acid must be added and the solution boiled in order to convert any *ferrous* salt present into a ferric salt. If iron is found, the original solution must then be tested to discover whether it was initially present as a ferrous or ferric salt.]

CHROMIUM, Cr . **Pp. Chromic Hydrate, $Cr_2(HO)_6$** (*dark green*).
ALUMINIUM, Al . „ **Aluminium Hydrate, $Al_2(HO)_6$** (*white*).

IRON (*Ferric*), **Fe‴**.

Solution—Ferric Chloride, Fe_2Cl_6.

1. Ammonium hydrate, AmHO gives a *reddish-brown* pp. of ferric hydrate, $Fe_2(HO)_6$.

2. Potassium ferrocyanide, K_4FeCy_6, gives a *dark blue* pp. of *prussian blue*, $Fe_4(FeCy_6)_3$.

3. Potassium ferricyanide, $K_6Fe_2Cy_{12}$, gives *no* pp., but a *deep green* (*very dilute solutions*) or *brown coloration*.

4. Potassium sulphocyanate, KCyS, gives the solution a blood-red colour on account of the formation of $Fe_2(CyS)_6$. The colour of the solution is readily destroyed by a solution of mercuric chloride, $HgCl_2$.

Ferric chloride Fe_2Cl_6 is the common soluble salt of Iron (Fe‴).

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* When AmCl and AmHO are both to be added to a solution, the AmCl should be added first, because it is often added to prevent the precipitation of hydrates by the AmHO.

IRON (*Ferrous*), **Fe″**.

Solution—Ferrous Sulphate, $FeSO_4$.

1. AmHO gives a *light coloured* pp. of ferrous hydrate, $Fe(HO)_2$, which rapidly turns *dirty green*, and more slowly *reddish-brown*, by reason of the absorption of oxygen: complete precipitation of ferrous hydrate by AmHO is prevented by the presence of ammonium salts.

2. Potassium ferrocyanide, K_4FeCy_6, gives a *bluish-white* pp., which speedily turns blue by absorption of oxygen.

3. Potassium ferricyanide, $K_6Fe_2Cy_{12}$, gives a *dark blue* pp. of $Fe_2Cy_6 3FeCy_2$ (*Turnbull's blue*). *Distinction from ferric salts.*]

4. Potassium sulphocyanate, KCys, produces neither a pp. nor change of colour when the solution is free from a ferric salt.

5. Ammonium sulphide, Am_2S, gives with both ferrous and ferric solution a black pp. of ferrous sulphide, FeS.

Ferrous sulphate, $FeSO_4$ (green vitriol), is the common soluble salts of iron, Fe″. Ferrous ammonium sulphate, $Fe''(NH_4)_2(SO_4)_2$, is a common double salt soluble in water.

CHROMIUM, **Cr.**

Solution—Chromic Chloride, Cr_2Cl_6.

1. AmHO gives a *dull green* pp. of chromic hydrate, $Cr_2(HO)_6$, with most chromium salts.

2. Potassium hydrate, KHO, and sodium hydrate, NaHO, give the same pp. soluble in excess of the alkali, and forming a *green* solution.

3. Chromic compounds may be recognised by converting the chromic oxide into chromic acid. This can be done in several ways—

(1) By boiling chromic chloride in solution with PbO_2 and NaHO or KHO. The reaction which occurs is accompanied by a *change of colour* from *green* to *yellow*, and may be expressed thus:—

$$\text{(i.)}\quad Cr_2Cl_6 + 6KHO = \underset{\text{Soluble in excess of KHO.}}{Cr_2(HO)_6} + 6KCl.$$

$$\text{(ii.)}\quad Cr_2(HO)_6 + 3PbO_2 = \underbrace{2PbCrO_4 + PbO}_{\text{Soluble in KHO.}} + 3H_2O.$$

When the solution is acidulated with acetic acid, a pp. of plumbic chromate, $PbCrO_4$, is obtained.

(2) By boiling a solution of Cr_2Cl_6 with NaHO, and sodic hypochlorite, NaOCl—

$$Cr_2(HO)_6 + 3NaOCl + 4NaHO = 2Na_2CrO_4 + 3NaCl + 5H_2O.$$

(**3**) By fusion with alkaline carbonates and nitre. Chromium compounds, when mixed with fusion mixture and nitre, and fused on platinum-foil, give a yellow mass of potassium and sodium chromate, which, when acidulated in solution with acetic acid, gives, with solutions of lead, a yellow pp. of $PbCrO_4$.

4. Chromium compounds give an emerald green borax bead, both in the inner and outer flame.

Chromous chloride, $CrCl_2$, and chromic chloride, Cr_2Cl_6, are the most important compounds in which chromium occurs as a base. *Chrome alum*, $KCr(SO_4)_2$, is a common soluble double salt. The *chromates* contain chromium as chromic acid (p. 68).

ALUMINIUM, **Al.**

Solution—Alum, $K_2Al_2(SO_4)_4$.

1. AmHO gives a *white transparent gelatinous* pp. of aluminium hydrate, $Al_2(HO)_6$.

2. Sodium hydrate, NaHO, or potassium hydrate, KHO, precipitates the hydrate: readily soluble in excess, re-precipitated by excess of AmCl. [*Distinction from zinc.*]

3. Hydric di-sodic phosphate, Na_2HPO_4, gives a *white bulky* pp. of aluminium phosphate, insoluble in AmHO and AmCl.

4. Heat on charcoal before the blowpipe: a white mass is obtained which is highly luminous while hot. Moisten with cobaltous nitrate, and heat again in the inner flame: a *fine blue mass* is obtained. [*Distinction from zinc.*]

The commonest substance containing aluminium is common (potash) alum, which is a soluble double sulphate of potassium and aluminium $KAl(SO_4)_2$. Ammonia-alum, $NH_4Al(SO_4)_2$ is another common alum.

GROUP IV.

METALS PRECIPITATED AS SULPHIDES BY AMMONIUM SULPHIDE, Am_2S, IN PRESENCE OF AmHO AND AmCl.

Group-reagent—AmCl, AmHO, and Am_2S.

NICKEL, Ni . . .	**Pp. Nickel Sulphide, NiS** (*black*).
COBALT, Co . . .	**„ Cobalt Sulphide, CoS** (*black*).
MANGANESE, Mn .	**„ Manganese Sulphide, MnS** (*buff*).
ZINC, Zn	**„ Zinc Sulphide, ZnS** (*white*).

NICKEL, **Ni.**

Solution—Nickel Sulphate, $NiSO_4$.

[*Note that the colour of the solution is green. All aqueous solutions of Nickel salts are of a greenish colour.*]

1. Am_2S gives a *black* pp. of nickel sulphide, NiS: slightly soluble in excess, producing a dirty black liquid.

2. Sodium hydrate, NaHO, gives an apple-green pp. of $Ni(HO)_2$: insoluble in excess of sodium hydrate.

3. Ammonia, AmHO, produces the same pp., which, however, dissolves in excess of the reagent to a blue liquid, and is not formed in presence of AmCl.

4. Potassium cyanide, KCy, gives a *yellowish green* pp. of nickel cyanide, $NiCy_2$: soluble in excess, giving a brownish yellow solution, containing a double cyanide of nickel and potassium, 2KCy, $NiCy_2$. On adding a strong solution of sodium hypochlorite, NaOCl, to this solution, a black pp. of $Ni_2(OH)_6$ is obtained *without heating*. [*Distinction from cobalt.*]

5. Heated on a borax bead, in the outer flame, compounds of nickel yield an intensely coloured bead, which appears *hyacinth-red* to *violet-brown* when *hot*, and *yellowish* to *sherry-red* when *cold*, according to the quantity of nickel present.

Nickel sulphate, $NiSO_4$, nickel chloride, $NiCl_2$, and nickel nitrate, $Ni(NO_3)_2$ are the commonest soluble nickel salts. They are all of a green colour and more or less deliquescent.

COBALT, Co.

Solution—Cobalt Nitrate, $Co(NO_3)_2$.

[*Note that the colour of the solution is a pale red. All aqueous solutions of cobalt salts are of a reddish colour.*]

1. Am_2S gives a *black* pp. of cobalt sulphide, CoS: insoluble in excess of Am_2S.

2. Potassium hydrate, KHO, or sodium hydrate, NaHO, gives a pp. of a *blue* basic salt, which on exposure to the air becomes *olive-green*, owing to the absorption of oxygen. When heated, a rose-red cobaltous hydrate, $Co(HO)_2$, is obtained, which contains usually a small quantity of dark brown cobaltic hydrate. The pp. is dissolved by ammonic carbonate to an intensely violet-red fluid.

3. AmHO gives a *bluish-green* pp., not formed in presence of AmCl.

4. Potassium cyanide, KCy, produces a *reddish-brown* pp. of cobaltous cyanide, $CoCy_2$, which is redissolved in excess of KCy, becoming, on boiling, potassium cobalticyanide, $K_6Co_2Cy_{12}$. This solution, when treated in the cold with strong sodium hypochlorite, NaOCl, gives *no* pp.

5. The borax bead is coloured *blue* in the outer and inner flame by cobalt compounds.

Cobaltous sulphate, $CoSO_4$, cobaltous nitrate, $Co(NO_3)_2$, and cobaltous chloride, $CoCl_2$, are the most important of the soluble cobalt salts.

MANGANESE, **Mn.**

Solution—Manganous Sulphate, $MnSO_4$.

1. Am_2S gives a *buff* or *flesh-coloured* pp. of manganese sulphide, MnS: readily soluble in dilute acids, even in acetic acid. [*Distinction from* Ni, Co, *and* Zn.]

2. Sodium hydrate, NaHO, or potassium hydrate, KHO, gives a *white* pp. of manganous hydrate, $Mn(OH)_2$, insoluble in excess. The pp. speedily absorbs oxygen from the air, and turns *dark brown* with the formation of the basic hydrate $(MnOHO)_2$, which is insoluble in AmCl.

3. Ammonia, AmHO, produces the same pp., except when ammonium salts are present, when the precipitation is prevented.

[On adding AmCl and AmHO to a solution of a manganese salt, a soluble double chloride of ammonium and manganese, 2AmCl, $MnCl_2$, is formed. On exposure to air, this solution is gradually oxidized, and the manganese precipitated as a dark brown di-hydrate, $(MnOHO)_2$. For this reason manganese may be precipitated in Group III., *but the precipitation does not take place until after exposure to the air for some time.* Ferric hydrate is precipitated *immediately*.]

4. Fuse a *very small* quantity of a manganese salt with fusion mixture and a little potassium nitrate, KNO_3, on a piece of platinum-foil. A *deep green mass* remains. This mass is a mixture of potassium and sodium manganates, K_2MnO_4 and Na_2MnO_4, and dissolves in water, giving a *pale pink* solution.

5. In the outer flame the borax bead is coloured *amethyst* when cold. In the inner flame the bead becomes nearly colourless.

The most important salts of manganese are manganous sulphate, $MnSO_4$ and manganous chloride, $MnCl_2$. Of these the sulphate is the more commonly met with. The manganic salts are very unstable.

ZINC, **Zn.**

Solution—Zinc Sulphate, $ZnSO_4$.

1. Am_2S gives a *white* pp. of zinc sulphide, ZnS.

2. Sodium hydrate, NaHO, precipitates zinc hydrate, $Zn(HO)_2$, as a *white gelatinous* pp., soluble in excess. Addition of AmCl does not reprecipitate the zinc hydrate. [*Distinction from aluminium.*]

3. Potassium ferrocyanide, K_4FeCy_6, gives a *white* pp. of Zn_2FeCy_6: insoluble in dilute acids.

4. Heat some of the dry salt on charcoal: a mass of zinc oxide, ZnO, remains, which is *yellow when hot*, and *white when cold.* Moisten it with a drop of cobaltous nitrate, $Co(NO_3)_2$, and again heat it. The mass now becomes *green.*

The more commonly occurring zinc salts are the sulphate (*white vitriol*, $ZnSO_4, 7H_2O$), the chloride, $ZnCl_2$, and the acetate $Zn(C_2H_3O_2)_2$.

GROUP V.

METALS PRECIPITATED AS CARBONATES BY AMMONIUM CARBONATE, Am_2CO_3, IN THE PRESENCE OF AmCl AND AmHO.

Group-reagent—AmCl, AmHO, and Am_2CO_3.

BARIUM, Ba . .	**Pp.**	**Barium carbonate, $BaCO_3$**	*(white).*
STRONTIUM, Sr .	,,	**Strontium carbonate, $SrCO_3$**	,,
CALCIUM, Ca . .	,,	**Calcium carbonate, $CaCO_3$**	,,

BARIUM, **Ba**.

Solution—Barium Chloride, $BaCl_2$.

1. Ammonium Carbonate, Am_2CO_3 gives a *white* pp. of barium carbonate.

2. Dilute sulphuric acid, H_2SO_4, and all soluble sulphates, give immediately a *heavy white granular* pp. of barium sulphate, $BaSO_4$, even from very dilute solutions of baric salts: insoluble in dilute acids and alkalies.

Solutions of strontium or calcium sulphate—two sulphates which are but slightly soluble in water—constitute the most delicate test for barium.

3. Potassium chromate, K_2CrO_4, gives a *lemon-yellow* pp. of barium chromate, $BaCrO_4$: soluble in nitric and hydrochloric acids, but insoluble in acetic acid. [*Distinction from strontium and calcium salts,* which give no pp. from dilute solutions.]

4. Hydrofluosilicic acid, H_2SiF_6, produces a *colourless crystalline* pp. of barium silicofluoride, $BaSiF_6$, which subsides quickly, especially when an equal bulk of alcohol is added: insoluble in alcohol; slightly soluble in water and dilute acids. [*Distinction of barium from strontium and calcium salts,* which give no pp.]

5. Barium compounds, when heated in the lower part of a Bunsen flame, colour the upper part of the flame yellowish green.

Barium Chloride $BaCl_2$ and barium nitrate $Ba(NO_3)_2$ are the commonest soluble salts of barium.

STRONTIUM, **Sr.**

Solution—Strontium Chloride, $SrCl_2$.

1. Ammonium Carbonate, Am_2CO_3 gives a *white* pp. of strontium carbonate, $SrCO_3$.

2. Dilute sulphuric acid, H_2SO_4, and all soluble sulphates produce a *white* pp. of strontium sulphate, $SrSO_4$. In dilute solutions a pp. appears after some time, more especially when calcium sulphate is used as the precipitant: heat favours the precipitation.

3. Potassium chromate, K_2CrO_4, and hydrofluosilicic acid, H_2SiF_6, give no pp. with strontium salts. [*Cf.* tests 3 and 4 for barium.]

4. Strontium compounds colour the flame an *intense crimson.*

Strontium nitrate $Sr(NO_3)_2$ and Chloride $SrCl_2$ are the commonest soluble compounds of strontium.

CALCIUM, **Ca.**

Solution—Calcium Chloride, $CaCl_2$.

1. Ammonium Carbonate, Am_2CO_3 gives a *white* pp. of calcium carbonate, $CaCO_3$.

2. Ammonium oxalate, $Am_2C_2O_4$, gives a *white powdery* pp. of calcium oxalate even in largely diluted solutions. The pp. is insoluble in acetic and oxalic acids, but is readily soluble in nitric and hydrochloric acids.

3. Dilute sulphuric acid or soluble sulphates give, *from concentrated solutions*, a white pp. of calcium sulphate, $CaSO_4$, which is slightly soluble in water.

[$CaSO_4$ gives no pp. with calcium compounds. This reagent is conveniently used to distinguish between Ba, Sr, and Ca. With Ba it gives *at once* a heavy white pp., with the Sr, the pp. is formed *only after some time*, and with Ca it is *not formed at all.*]

4. Potassium chromate, K_2CrO_4, and hydrofluosilicic acid, H_2SiF_6, give no pp. with calcium salts.

5. Calcium compounds colour the flame a yellowish red.

The Chloride and Sulphate are the compounds of calcium most commonly met with.

GROUP VI.

No Group-reagent.

MAGNESIUM, Mg.	**SODIUM, Na.**
POTASSIUM, K.	**AMMONIUM, NH_4** (Am).

MAGNESIUM, **Mg.**

Solution—Magnesium Sulphate, $MgSO_4$.

1. Ammonia, AmHO, *in the presence of* AmCl gives *no* pp., because of the formation of the soluble double chloride of ammonium and magnesium, $2AmCl.MgCl_2$. *In the absence of* AmCl, part of the magnesia is precipitated as the hydrate $Mg(HO)_2$, the double chloride being also formed at the same time, thus:

$$2MgCl_2+2AmHO = Mg(HO)_2+2AmCl.MgCl_2.$$

[The double chloride formed in the presence of AmCl is not precipitated by $AmCO_3$; hence magnesium is not precipitated in Group V., provided a sufficient quantity of AmCl be present.

2. Hydrogen disodium phosphate, Na_2HPO_4, in the presence of AmHO and AmCl, gives a *white crystalline* pp. of ammonium magnesium phosphate, $MgAmPO_4$. The separation from a dilute solution of a salt of magnesium is promoted by heating gently and stirring with a glass rod. Very dilute solutions give a precipitate only after standing some hours in a warm place.

3. Baryta water and lime water precipitate the hydrate $Mg(HO)_2$ when ammonium salts are not present.

4. Heat strongly before the blowpipe on charcoal: moisten residue with cobaltous nitrate, and again heat: a *pale pink mass* will be obtained. [*Distinction from zinc and aluminium.*]

Magnesium sulphate $MgSO_4$ and magnesium chloride are the commonest soluble magnesium compounds.

POTASSIUM, **K.**

Solution—Potassium Chloride, KCl.

1.* Place a few drops of the solution on a watch-glass, and add to it two or three drops of platinic chloride, $PtCl_4$, a little HCl, and then a few drops of alcohol. If the solution is concentrated, a *dense yellow crystalline* pp. of the double chloride, $PtCl_4 . 2KCl$, is at once formed; if the solution be dilute, the pp. is obtained only after some time. In this case, if allowed to stand quietly for half-an-hour or so, a yellow crystalline pp. forms on the bottom of the glass, where it is readily detected by stirring it with the point of a thin glass rod. In very dilute solutions no pp. is obtained.

2. Tartaric acid gives in concentrated neutral or slightly acid solutions a *white crystalline* pp. of cream of tartar. Shaking or stirring favours the precipitation.

3. Potassium compounds colour the flame *violet*. Owing to the presence of traces of sodium in all potassium salts, this flame test is not always distinct. By looking at the flame through a piece of blue cobalt glass the potassium flame can be distinguished in the presence of sodium, but this method is not very reliable, and, unless the potassium flame is very distinct, its presence will need careful confirmation in the wet way.

The nitrate, chloride, bromide, iodide, carbonate, and sulphate are the most common potassium compounds.

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* Potassic iodide, KI, is not precipitated by $PtCl_4$ owing to the formation of a *soluble* double salt.

SODIUM, **Na.**

1. Platinic chloride, $PtCl_4$, and tartaric acid give *no* pp.

2. Potassium metantimoniate, $KSbO_3$, gives a *white crystalline* pp. of sodic metantimoniate, $NaSbO_3$, from *concentrated* neutral or alkaline solutions.

3. Sodium compounds colour the flame an *intense golden yellow*.

[This test is the most reliable for sodium, but it must be remembered that traces of sodium are always present, so that a faint yellow coloration of the flame may in general be neglected. When sodium is present in quantity, the colour is characteristic and *very* intense.]

All sodium compounds are soluble in water, the most common being the chloride, sulphate, nitrate, carbonate, and phosphate.

AMMONIUM, **NH_4(Am)**.

Solution—Ammonium Chloride, AmCl.

1. Platinic chloride, $PtCl_4$, produces in neutral and concentrated acid solutions a *dense yellow* pp. of the double chloride, $PtCl_4 . 2AmCl$.

2. Add NaHO or KHO, and heat gently. Ammonia, NH_3, is given off, and is readily recognised by smell, action on litmus paper, and by fuming when a glass rod dipped in HCl is brought near it.

3. Heat some dry ammonium chloride in a test tube: a white sublimate is formed in the upper part of the tube. All ammonium salts are volatilized or decomposed by heat.

4. *Nessler's test* (potassic mercuric iodide) gives a *brown* pp. of $NHg_2I.OH_2$, or a *yellowish brown coloration*, according to the amount of ammonia present. This reaction is too delicate for the analysis of ordinary salts, and should only be used for detecting traces of ammonia.

The most common salts of ammonium are the chloride, carbonate, sulphate, bromide, and nitrate.

CHAPTER III.

REACTIONS OF THE ACIDS.

The acids do not admit of classification into groups.

SULPHURIC ACID, $\mathbf{H_2SO_4}$.

Solution—Potassium Sulphate, $\mathbf{K_2SO_4}$.

1. Barium chloride, $BaCl_2$, gives a *heavy white powdery* pp. of barium sulphate, $BaSO_4$, insoluble in HCl.

Use Barium Nitrate $Ba(NO_3)_2$ if Silver, Mercury (-ous), or Lead is present.

2. Lead acetate, $Pb(C_2H_3O_2)_2$, gives a *heavy white* pp. of lead sulphate, $PbSO_4$.

SULPHUROUS ACID, $\mathbf{H_2SO_3}$.

Solution—Sodium Sulphite, $\mathbf{Na_2SO_3}$.

1. Add HCl, and, if necessary, heat: the smell of burning sulphur is perceptible, SO_2 being evolved *without* the precipitation of sulphur.

2. To some pieces of granulated zinc in a test tube add a little hydrochloric acid till a brisk effervescence takes place; then pour in some of the liquid to be tested, and hold a piece of filter paper moistened with lead acetate in the escaping gas. Any sulphite present will be reduced by the hydrogen and converted into sulphuretted hydrogen, H_2S, which will stain the paper black with lead sulphide.

$$H_2SO_3 + 3H_2 = H_2S + 3H_2O.$$

3. Barium chloride, $BaCl_2$, gives a *white* pp. of $BaSO_3$, *soluble* in HCl. Most sulphites contain small quantities of sulphate (from oxidation) and hence the pp. with $BaCl_2$ is usually not completely soluble in HCl.

HYPOSULPHUROUS ACID (*Thiosulphuric Acid*), $\mathbf{H_2S_2O_3}$.

Solution—Sodium Hyposulphite (Thiosulphate), $\mathbf{Na_2S_2O_3}$.

1. Hydrochloric or dilute sulphuric acid gives, on warming, a smell of SO_2, and a *whitish yellow* pp. of sulphur.

2. Silver nitrate, $AgNO_3$, added gradually, in excess, gives, after some time, a *white* pp., $Ag_2S_2O_3$. The pp. slowly decomposes, becoming *yellowish brown* and then *black*, forming silver sulphide, Ag_2S, thus—

$$Ag_2S_2O_3 + H_2O = Ag_2S + H_2SO_4.$$

HYDROSULPHURIC ACID, $\mathbf{H_2S}$.

Solution—Ammonium Sulphide, $\mathbf{Am_2S}$.

1. Heat with HCl: sulphuretted hydrogen gas is given off, which is known by its characteristic odour and the black stain it produces on paper moistened with lead acetate; and most soluble sulphides, since they contain persulphides, give a white pp. of sulphur.

2. Lead acetate gives a *black* pp. of lead sulphide, PbS.

3. Soluble cadmium salts give a *fine yellow* pp. of cadmium sulphide, CdS.

4. Heat a dry sulphide in a piece of glass-tubing held obliquely over a flame: in general, sulphur dioxide gas, which may be recognised by its odour, is given off.

Tests 2—4 are applicable to the detection of H_2S as an aqueous solution or as a soluble sulphide.

Note.—All salts of sulphur acids may be detected thus. Fuse with Na_2CO_3 on charcoal in the inner blowpipe flame, place the mass on a bright silver coin, and moisten it with water: a *black stain* will be produced on the silver. If the mass be acidified, sulphuretted hydrogen is given off.

HYDROCHLORIC ACID, **HCl.**

Solution—Sodium Chloride, NaCl.

1. Silver nitrate, $AgNO_3$, gives a *white curdy* pp. of silver chloride, AgCl: insoluble in strong boiling nitric acid (silver cyanide, AgCy, dissolves): soluble in solution of ammonia.

2. Mix with MnO_2* and H_2SO_4, and heat: chlorine gas is given off, and may be recognised by its colour, odour, and bleaching action.

3. Strong sulphuric acid, when added to a strong solution of a chloride or to the dry salt, produces acid fumes of HCl.

CHLORIC ACID, **$HClO_3$.**

Solution—Potassium Chlorate, $KClO_3$.

1. Heat a small quantity of the dry salt in a test tube: fusion first takes place, then oxygen is evolved, and when action ceases a residue of potassium chloride, KCl, remains. Dissolve this residue in water, and test for HCl by 1, 2, and 3, above.

2. On adding strong H_2SO_4 to a few small crystals of $KClO_3$ in a test tube, a yellowish gas, ClO_3, is evolved. On gently heating, a series of detonations occur.

3. On heating the dry salt, oxygen is evolved, and may be detected by means of a glowing splinter. On charcoal it deflagrates like a nitrate, but more violently.

Note.—Most chlorates are soluble in water; hence the absence of tests in the wet way.

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* Common MnO_2 frequently contains chlorine, the best is "needle" or native "manganese."

HYPOCHLOROUS ACID, **$HClO$.**

The hypochlorites are all soluble in water, and are found only in combination or mixture with the chlorides of the same metal. Bleaching powder, $Ca(OCl)Cl$, is the most familiar example of such a compound. Hypochlorites are distinguished by their powerful bleaching action in the presence of acids.

1. Shake up some bleaching powder with water, and filter off; then add to the filtrate dilute sulphuric acid, H_2SO_4: chlorine is evolved, and may be recognised by colour, odour, and bleaching action.

HYDROBROMIC ACID, **HBr.**

Solution—Potassium Bromide, KBr.

1. Silver nitrate, $AgNO_3$, gives a *yellowish white* pp. of silver bromide, $AgBr$: insoluble in dilute nitric acid; slightly soluble in ammonia.

2. Add chlorine water to the solution: the liquid becomes yellow from free bromine. Shake up with some carbon disulphide, CS_2, which does not mix with the solution, but dissolves out the bromine, and an *orange-coloured* solution of bromine in CS_2 sinks to the bottom.

3. Mix with MnO_2 and H_2SO_4, and heat: bromine vapour of a *reddish brown* colour is evolved, which colours paper moistened with starch, *yellow*. [*Distinction from iodine.*]

HYDRIODIC ACID, **HI.**

Solution—Potassium Iodide, KI.

1. Silver nitrate, $AgNO_3$, gives a yellow pp. of silver iodide, AgI: insoluble in nitric acid, and nearly insoluble in solution of ammonia.

2. Mercuric chloride, $HgCl_2$, gives a pp., yellow changing to *scarlet*, of mercuric iodide, HgI_2: soluble in excess of $HgCl_2$ or of KI.

3. Add a *few drops* of chlorine water, the solution becomes *brown* from liberation of iodine. Shake up with a little CS_2: a fine violet-coloured solution of iodine in CS_2 sinks to the bottom. [*If too much chlorine water is used, colourless* ICl_3 *is formed.*]

4. Mix with MnO_2 and H_2SO_4, and heat in a long test tube: *violet*-coloured iodine vapour is given off, which condenses in the cool part of the tube in grey-coloured crystals, and turns paper moistened with starch paste *blue.*

HYDROFLUORIC ACID, **HF.**

Most fluorides are insoluble in water. The only one of importance is *fluor spar*, calcium fluoride, CaF_2.

Place some powdered fluor spar in a small leaden trough; add strong H_2SO_4 and mix to a thin paste. HF will now be freely evolved, and if a sheet of glass coated with wax be written on with a sharp point, and then placed, writing downwards, on the trough, the writing will be etched on the glass by the action of the fumes of HF on the exposed glass.

HYDROCYANIC ACID, **HCN** (HCy).

Solution—Potassium Cyanide, KCy.

1. Silver nitrate, $AgNO_3$, gives a *white* pp. of silver cyanide, AgCy, which is at first redissolved, but remains when an excess of $AgNO_3$ has been added. Wash and dry the pp., and then heat to redness. Metallic silver remains. [Silver chloride, AgCl, melts when heated, but is not decomposed.]

2. Dilute sulphuric acid, H_2SO_4, when added to a very small quantity of KCN, gives the peculiar odour of hydrocyanic acid, HCy.
[N.B.—This vapour is very poisonous.]

3. Ferrous sulphate, $FeSO_4$, and ferric chloride, Fe_2Cl_6, are added, then sodium hydrate, and lastly excess of HCl, a *deep blue* pp. of prussian blue is produced.

4. Place a little of the solution in a small beaker, add a few drops of dilute sulphuric acid, cover the beaker with a watch-glass on the lower side of which a drop of yellow ammonium sulphide has been placed, and gently warm the beaker on a sand tray. After a few minutes, remove the watch-glass and evaporate the drop of ammonium sulphide upon its lower surface to dryness over the flame, taking care not to overheat it; when cold, moisten the residue with a drop of ferric chloride solution: a blood-red coloration will be produced $Fe_2(CyS)_6$. Simply evaporating a portion of the original solution with a little Am_2S on the water-bath, and when dry adding Fe_2Cl_6, will, in many cases, be quite sufficient. This is one of the best tests for hydrocyanic acid.

HYDROFERROCYANIC ACID, $\mathbf{H_4FeCy_6}$.

Solution—Potassium Ferrocyanide, $\mathbf{H_4FeCy_6}$.

1. Silver nitrate gives a white pp. of Ag_4FeCy_6, insoluble in nitric acid, insoluble in ammonia.

2. Ferrous sulphate, $FeSO_4$, free from ferric salt, gives a *white* pp., which speedily turns *blue* when exposed to the air.

3. Ferric chloride, Fe_2Cl_6, gives a pp. of *prussian blue*.

4. Copper sulphate, $CuSO_4$, gives a *reddish brown* pp. of cupric ferrocyanide.

HYDROFERRICYANIC ACID, $\mathbf{H_6Fe_2Cy_{12}}$.

Solution—Potassium Ferricyanide, $\mathbf{K_6Fe_2Cy_{12}}$.

(This is the only common ferricyanide.)

1. Ferrous sulphate, $FeSO_4$, gives a *deep blue* pp. (*Turnbull's blue*).

2. Ferric chloride, Fe_2Cl_6 gives *no* pp., but a *deep brown* or *greenish coloration.*

3. Silver nitrate gives an *orange-red* pp. of $Ag_6Fe_2Cy_{12}$, soluble in ammonia.

NITRIC ACID, $\mathbf{HNO_3}$.

Solution—Potassium Nitrate, $\mathbf{KNO_3}$.

1. Take a weak solution of the nitrate, and dissolve in it a crystal of ferrous sulphate, $FeSO_4$. When quite cold, pour into the tube enough concentrated H_2SO_4 to form a distinct layer at the bottom. A deep purple or brown-coloured ring will appear where the liquids meet.

Note.—When the addition of $FeSO_4$ produces a pp. (*e.g.*, with barium nitrate), a portion of the solution should be boiled with sodium carbonate, then filtered, concentrated, and the filtrate acidified with dilute H_2SO_4. The test may then be applied to this solution which contains the acid as sodium nitrate, the base having been precipitated as a carbonate.

2. Heat with concentrated sulphuric acid and copper turnings: nitric oxide, NO, is given off, which becomes nitric peroxide, N_2O_4, in the upper part of the tube from absorption of oxygen.

3. A nitrate heated on charcoal deflagrates.

Note.—All nitrates are soluble in water; hence we have no precipitation reactions. Compare chloric acid.

NITROUS ACID, $\mathbf{HNO_2}$.

Solution—Potassium Nitrite, $\mathbf{KNO_2}$.

1. On addition of dilute sulphuric acid, H_2SO_4, a yellow gas, N_2O_3, is given off. No gas is evolved when nitrates are mixed with dilute sulphuric acid.

2. A solution of ferrous sulphate gives a *dark brown coloration* on the addition of *dilute* sulphuric acid. [*Distinction from* HNO_3.]

3. A dilute solution of potassic iodide, KI, acidified with H_2SO_4, and to which some starch paste has been added, gives immediately the *blue* iodide of starch.

CARBONIC ACID, $\mathbf{H_2CO_3}$.

1. All carbonates are decomposed by dilute acids with the evolution of CO_2. This may be detected by suspending a drop of lime water on a glass tube over the gas, when the drop soon turns milky.

Note.—Acid carbonates, or bi-carbonates, evolve CO_2 on boiling, and are converted into carbonates ; *e.g.*,

$$2KHCO_3 = K_2CO_3 + CO_2 + H_2O.$$

2. Add $NaHCO_3$ to a solution of $MgSO_4$: no pp. is formed, but on boiling the solution a white pp. of $MgCO_3$ is quickly formed. The normal carbonate, Na_2CO_3, gives a pp. of $MgCO_3$ at once.

BORIC ACID, $\mathbf{H_3BO_3}$.

Solution—Borax, $\mathbf{Na_2B_4O_7}$.

1. Add HCl to a solution of borax ; wet a piece of turmeric paper with the solution. The paper when dried becomes reddish brown, and, on treating with *dilute* caustic soda, changes to a purplish blue-black.

Dry the paper by coiling round the exterior of a test-tube, partially filled with water, and boiling the water in the tube over the Bunsen flame.

2. Place a small quantity of the dry salt in a porcelain dish, and make into a paste with H_2SO_4 ; cover with alcohol, and ignite : the alcohol burns with a *green flame.*

3. Place a small quantity of the dry salt on the loop of the platinum-wire, and heat in the Bunsen flame. The borate at once swells up, and then shrinks and fuses to a transparent glass. If the salt is moistened with HCl or H_2SO_4 before heating, the flame is tinged *green.*

SILICIC ACID, H_4SiO_4.

Solution—Sodium Silicate, Na_4SiO_4.

1. Add HCl, and evaporate in an evaporating dish to dryness; when cold, moisten with HCl, heat, and add water: white flakes of silica, SiO_2, will remain undissolved.

2. Fuse a colourless silicate in a bead of microcosmic salt: the silica floats about in the bead as a skeleton.

PHOSPHORIC ACID, H_3PO_4.

Solution—Sodium Phosphate, Na_2HPO_4.

1. Silver nitrate, $AgNO_3$, gives a *yellow* pp. of silver phosphate, Ag_3PO_4, soluble in AmHO and in acids.

2. Ammonium chloride, AmCl, ammonia, AmHO, and magnesium sulphate, $MgSO_4$, give a *white crystalline* pp. of ammonium magnesium phosphate, $AmMgPO_4$. Precipitation is favoured in dilute solutions by agitation and allowing to stand for a time.

3. Ammonium molybdate, $AmHMoO_4$, gives, on gently warming, a *finely divided yellow* pp. in solutions containing excess of nitric acid. In dilute solutions the liquid becomes yellow, but a pp. is formed only after gentle warming and standing for some hours. The solution must not be too strong in phosphate.

4. Barium chloride, $BaCl_2$, gives a *white* pp. of $Ba_3(PO_4)_2$, soluble in dilute acids.

ARSENIC ACID, $\mathbf{H_3AsO_4}$.

Solution—Sodium Arsenate, $\mathbf{Na_2HAsO_4}$.

1. Silver nitrate, $AgNO_3$, gives a *brick-red* pp. of silver arsenate, Ag_3AsO_4. [*Distinction from phosphoric acid.*]

2. Ammonium chloride, AmCl, ammonia, AmHO, and magnesium sulphate, $MgSO_4$, produce a *white* crystalline pp. of $AmMgAsO_4$, similar in appearance and properties to $AmMgPO_4$.

3. Ammonium molybdate, $AmHMoO_4$, in excess gives, on boiling, a *yellow* pp.

4. Add concentrated hydrochloric acid; heat to boiling, and pass H_2S a *yellow* pp. of arsenic sulphide, As_2S_3, mixed with sulphur is obtained. [*Difference from phosphate.*]

The reduction proceeds much more quickly if sulphurous acid be added before passing H_2S.

ARSENIOUS ACID, $\mathbf{H_3AsO_3}$.

Solution—Arsenious Oxide, $\mathbf{As_4O_6}$, dissolved in Hydrochloric Acid, $\mathbf{HCl}$.

[*See Arsenic, page* 40.]

CHROMIC ACID, $\mathbf{H_2CrO_4}$.

Solution—Potassium Chromate, $\mathbf{K_2CrO_4}$.

(All chromates are red or yellow.)

1. Silver nitrate, $AgNO_3$, gives a *dark red* pp. of silver chromate, Ag_2CrO_4.

2. On adding dilute sulphuric acid, the colour of the solution darkens, owing to formation of chromic acid, H_2CrO_4. To this solution add sulphurous acid: the solution becomes *green* from reduction, thus:—

$$2H_2CrO_4 + 3H_2SO_3 = Cr_2(SO_4)_3 + 5H_2O.$$

3. Lead acetate gives a *yellow* pp. of lead chromate, $PbCrO_4$.

4. On passing a current of H_2S gas through an acid solution of a chromate, reduction takes place; the solution becomes green owing to formation of chromic chloride, Cr_2Cl_6, and sulphur is precipitated. Thus

$$2K_2CrO_4 + 3H_2S + 10HCl = 4KCl + Cr_2Cl_6 + 8H_2O + 3S.$$

Note.—This reaction should be carefully noted, as it is likely to cause perplexity in testing for Group II.

CHAPTER IV.

SYSTEMATIC ANALYSIS.

Preliminary Examination in the Dry Way.

If the substance is in solution, a portion should be evaporated to dryness on a water-bath while the examination in the wet way is being proceeded with. The residue after evaporation should then be examined as below, I.–VI.

I. Note the general physical characteristics of the substance.

On this point the previous experience of the student must be his guide, and for this purpose he should note the appearance, colour, lustre, crystalline form, &c. of all substances with clearly marked characteristics which come under his notice. It is useless to give any list of such subtances, as they must be seen to be recognised.

II. Heat a small portion of the powdered substance in a bulb-tube in the Bunsen flame.

Observation.	Inference.
1. *The substance fuses and solidifies on cooling.*	Salts of the alkalies or alkaline earths.
2. *The substance changes colour—*	
(*a*) *Yellow* while *hot*, *white* on *cooling*.	ZnO.
(*b*) From *white* to *yellowish brown* when *hot*, *dirty light yellow* on *cooling*.	SnO_2.
(*c*) *Dark red* while *hot*, *yellowish brown* on *cooling*.	Fe_2O_3.
(*d*) From *white* to *orange* and *reddish brown*, *dull yellow* on cooling, fusible at an intense heat.	Bi_2O_3.
(*e*) From *yellow* to *reddish brown* while *hot*, *yellow* when *cold*, fusible at a red heat.	PbO.
3. *The substance gives off water with or without fusion.*	Salts containing water of crystallization.
4. *The substance swells up and shrinks.*	Alums, phosphates, borates.

II.—*continued.*

OBSERVATION.	INFERENCE.
5. *The substance volatilizes and condenses as a sublimate in the upper part of the tube*—	S, Hg, As, Sb, NH_4.
(*a*) *Reddish* drops, *yellow* when *cold.*	Sulphur or a sulphide.
(*b*) *Black* sublimate, which becomes *red* on rubbing.	HgS.
(*c*) *Yellow* sublimate.	As_2S_3, HgI_2.
(*d*) Sublimate of *small white octahedral crystals.*	As_2O_3.
6. *A gas is evolved*—	
(*a*) O (test with glowing splinter).	Chlorates, nitrates, peroxides.
(*b*) Brown fumes of N_2O_4.	Nitrates of heavy metals, Pb, Bi, Hg, &c.
(*c*) NH_3 (smell and alkaline reaction).	Salts of NH_4, combined with a *non-volatile acid.*
(*d*) Violet vapours of iodine.	Iodides.
(*e*) CO_2 (lime-water test).	Carbonates (except those of the alkalies).
(*f*) Cl (bleaching power, smell and colour).	Hypochlorites and some chlorides and chlorates.
(*g*) SO_2 (smell).	Sulphites, sulphur compounds.
(*h*) $(CN)_2$ (smell, rose-coloured flame).	Cyanides of heavy metals.

III. HEAT A SMALL QUANTITY OF THE SUBSTANCE IN A TEST-TUBE WITH A LITTLE DILUTE HCl.

OBSERVATION	INFERENCE.
1. *A gas is evolved*—	
(*a*) CO_2 (lime-water test).	Carbonates.
(*b*) H_2S (smell, and lead acetate paper test).	Sulphides, except those of the metals of Group II.
(*c*) SO_2 (smell of burning sulphur).	H_2SO_3.
(*d*) SO_2 and separation of yellow sulphur.	$H_2S_2O_3$.
(*e*) Cl (smell, and bleaching test).	HClO or oxidizing agents, *e.g.*, chromates, chlorates, &c.
(*f*) HCy (smell of bitter almonds).	Cyanides.

IV. HEAT A PORTION OF THE SUBSTANCE ON CHARCOAL IN THE INNER BLOWPIPE FLAME.

OBSERVATION.	INFERENCE.
1. *It decrepitates.*	NaCl, KI, &c.
2. *It fuses and sinks into the charcoal.*	Alkaline salts.
3. *It deflagrates.*	Chlorates, nitrates.
4. *A white mass remains, highly luminous while heated.*	Alkaline earths, Al_2O_3, SiO_2, ZnO.
5. *The cold residue of 4 when moistened with cobaltous nitrate,* $Co(NO_3)_2$ *and strongly heated, is coloured.*	
Blue.	Al_2O_3, phosphates and silicates.
Pale pink (colour indistinct).	MgO.
Green.	ZnO, Sb.
6. *A coloured residue is left on the charcoal.*	Cu, Co, Ni, Mn, Fe, Cr.
7. *An incrustation is formed on the charcoal—*	
(*a*) *White* incrustation.	Am, Hg.
(*b*) *White* (with garlic odour).	As.
(*c*) *Yellow* when *hot*, *white* when *cold.*	Zn.
(*d*) *Reddish brown.*	Cd.

V. MIX A PORTION OF THE SUBSTANCE CAREFULLY WITH FUSION MIXTURE AND A TRACE OF KCy, AND HEAT ON CHARCOAL UNDER THE INNER BLOWPIPE FLAME.

OBSERVATION.	INFERENCE.
1. *A metallic bead is obtained.*	
(*a*) A white malleable bead which marks paper (*yellow incrustation* on charcoal).	Pb.
(*b*) Soft, white malleable bead (*slight yellowish incrustation*).	Sn.
(*c*) Hard white malleable bead (*no incrustation*).	Ag.
(*d*) White brittle bead (*yellow incrustation*).	Bi.
(*e*) White brittle bead (*white fumes* and *incrustation*).	Sb.
2. *Red infusible scales.*	Cu.
3. *Magnetic metallic particles.*	Fe, Ni, Co.
4. *When the fused mass is placed on a bright silver coin and moistened with a drop of water; a black stain of* Ag_2S *is produced.*	Sulphur compounds.

VI. If the substance is coloured, heat on borax bead.

Observation.		Inference.
Oxidizing Flame.	*Reducing Flame.*	
Green.	Green.	Cr.
Yellow (cold), brown (hot).	Dirty green, olive.	Fe.
Amethyst.	Colourless.	Mn.
Violet (hot) to yellow-brown (cold).	Grey and opaque.	Ni.
Blue.	Blue.	Co.
Green (hot), blue (cold).	Colourless or red.	Cu.
Black (too much of the substance has been used).		

VII. Hold a piece of the substance moistened with HCl on the loop of a *CLEAN* platinum wire in the Bunsen flame.

1. The	flame	is	*yellow,*	Na.
2.	,,	,,	*violet,*	K.
3.	,,	,,	*intense crimson,*	Sr.
4.	,,	,,	*yellowish-green,*	Ba.
5.	,,	,,	*orange-red,*	Ca.
6.	,,	,,	*green,*	HBO_3 or Cu.
7.	,,	,,	*blue,*	Pb, Bi, $CuCl_2$, As_4O_6.

It is almost impossible to distinguish from one another, by the eye, the colours given to the flame by Pb, Bi, As, Sb, and K. Arsenic compounds always evolve a visible white fume, and lead invariably corrodes the platinum.

EXAMINATION FOR BASE.

Preparation of Solution.

1. If the substance is solid, it should be finely powdered, then try if it is soluble in water, in the cold or on boiling. If insoluble in water, try first dilute HCl, then concentrated HCl. If insoluble in HCl, try *aqua regia*.

Note. — It will generally be found unnecessary to employ *aqua regia*, but, if used, the solution should be evaporated to dryness with excess of HCl, and the residue dissolved in warm water before proceeding with the examination.

2. If the substance is insoluble in the above solvents, powder it very finely and mix thoroughly with five or six times its weight of pure fusion mixture. Heat the mixture on a boat made of a large piece of platinum-foil, first over the Bunsen flame, and then over the blowpipe. When cold, boil the foil and the fused mass with water until the whole mass is completely broken up and separated from the foil. The *solution* now contains the *acid* of the substance combined with K and Na, while the *residue* contains the *base* as an oxide or carbonate. Filter off and wash the residue with hot water until the washings give no alkaline reaction. Then—

(*a*) Test *filtrate* for *acid*, as directed in examination for acid.

(*b*) Test residue for base by dissolving in dilute HCl and proceeding as detailed below.

Note.—The most commonly occurring insoluble substances are—native or ignited alumina and chromic oxide; silica and many silicates; barium, strontium and lead sulphates; silver chloride, cyanide, bromide, and iodide; many minerals, *e.g.*, chrome iron ore, calcium fluoride; carbon and sulphur.

3. If the substance is provided in solution, or soluble in water, test with litmus paper. If the reaction is—

(*a*) *Acid.* Free acid, acid salt, or a normal salt having an acid reaction, may be present.

(*b*) *Alkaline.* Free alkali, or alkaline earth, or a cyanide or sulphide of an alkali, or a strong alkali (*e.g.*, KOH or NaOH united with a weak acid—*e.g.*, H_2CO_3, H_3PO_4, &c.), may be present.

(*c*) *Neutral.* Water, or one of a large number of salts, may be present.

When the solution is prepared, the examination must be carried on in accordance with the following tables. When the base is found by this method, its presence should be further confirmed by the special tests given in Chapter II.

TABLE A.

<table>
<tr><td colspan="6">GENERAL TABLE FOR THE ANALYSIS OF A SUBSTANCE CONTAINING ONLY ONE BASE.</td></tr>
<tr><td colspan="6">To a portion of the solution add **HCl.**</td></tr>
<tr><td rowspan="5">Pp., if produced,
may be—
Ag Cl (*white*).
$'\mathbf{Hg}'_2\mathrm{Cl}_2$ (*white*).
$\mathbf{Pb}\,\mathrm{Cl}_2$ (*white*).
If pp. is produced, continue analysis by Table I.
——
Note I.—If solution is dilute, Pb may not be precipitated in this group.
Note II. — Do not confound the precipitate of the oxychlorides of Antimony and Bismuth (see p. 36, 2 and p. 42, 2) with that of these chlorides.</td><td colspan="5">If no pp. is produced, to the *same* portion of the solution add $\mathbf{H_2S}$ water.
If a pp. is indicated, pass a current of $\mathbf{H_2S}$ gas through the solution.</td></tr>
<tr><td rowspan="4">Pp., if produced, may be—
Hg S (*black*)
Pb S (*black*)
$\mathbf{Bi}_2\mathrm{S}_3$ (*black*)
Cu S (*black*)
Cd* S (*yellow*)
Insoluble in NaHO or $\mathrm{Am_2S}$.
Sn S (*brown* or *black*)
$\mathbf{Sn}\,\mathrm{S}_2$ (*yellow*)
$\mathbf{As}_2\mathrm{S}_3$ (*yellow*)
$\mathbf{Sb}_2\mathrm{S}_3$ (*orange*)
Soluble in NaHO or $\mathrm{Am_2S}$.
If pp. is produced, continue analysis by Table II.
* Before passing on to next group, dilute the solution considerably with water, and gently warm; Cd may then be precipitated. The presence of much HCl prevents the precipitation of CdS.</td><td colspan="4">If no pp. is produced, to a fresh portion of the solution add **AmCl** and **AmHo** in excess, after boiling with a *few drops* of $\mathrm{HNO_3}$ to oxidize $\mathrm{Fe''}$ if present to $\mathrm{Fe'''}$. (See page 43)</td></tr>
<tr><td rowspan="3">Pp., if produced, may be—
$\mathbf{Fe}_2(\mathrm{OH})_6$ (*reddish-brown*).
$\mathbf{Cr}_2(\mathrm{OH})_6$ (*dark green*).
$\mathbf{Al}_2(\mathrm{OH})_6$ (*white*).
If pp. is produced, continue analysis by Table III.
——
A **phosphate** of any of the metals of Groups III., IV., and V., or Mg, might be precipitated by AmCl and AmHo.
Mn, if present, is also partially precipitated in this group. See Note to Table III.</td><td colspan="3">If no pp. is produced, to the same portion of the solution add $\mathbf{Am_2S}$.</td></tr>
<tr><td rowspan="2">Pp., if produced, may be—
Ni S (*black*).
Co S (*black*).
Mn S (*buff*).
Zn S (*white*).
If pp. is produced, continue analysis by Table IV.</td><td colspan="2">If no pp. is produced, to a fresh portion of the solution add **AmCl, AmHO** in excess, and $\mathbf{Am_2CO_3}$.</td></tr>
<tr><td>Pp., if produced, may be—
$\mathbf{Ba}\,\mathrm{CO}_3$ (*white*).
$\mathbf{Sr}\,\mathrm{CO}_3$ (*white*).
$\mathbf{Ca}\,\mathrm{CO}_3$ (*white*).
If pp. is produced, continue analysis by Table V.</td><td>If no pp. is produced by above reagents, base must be either
Mg,
$\mathbf{NH_4}$,
Na, or
K.
Continue analysis by Table VI.</td></tr>
</table>

TABLE I.

<table>
<tr><th colspan="3">Examination for the Metals of Group I.</th></tr>
<tr><td colspan="3">Shake up the pp., allow to subside, and pour off the clear liquid. To the residue add an excess of AmHO.</td></tr>
<tr><td>The pp. dissolves and is reprecipitated on addition of HNO_3 in excess.
Presence of **Ag.**</td><td>The pp. is blackened.
Presence of **'Hg'** (*mercurous*).</td><td>The pp. is not affected by AmHO.
Presence of **Pb.**
Confirm by precipitating a fresh portion of the solution with HCl. On boiling, the pp. should dissolve.</td></tr>
<tr><td colspan="3">Confirm by special tests on pp. 30 to 32.</td></tr>
</table>

TABLE II.

<table>
<tr><td colspan="8">EXAMINATION FOR THE METALS OF GROUP II.</td></tr>
<tr><td colspan="8">Filter and wash pp. on filter until free from HCl and H_2S, then gently warm a portion of the pp. with NaHO, or Am_2S.</td></tr>
<tr><td colspan="5">The pp. does not dissolve.</td><td colspan="3">The pp. dissolves. Heat another portion of the pp. with AmC_2O_3.</td></tr>
<tr><td colspan="4">The pp. is black. Boil a fresh portion of it with strong HNO_3 and a few drops of dilute H_2SO_4.</td><td rowspan="3">A <i>yellow</i> pp. indicates presence of Cd.</td><td rowspan="3">The <i>yellow</i> pp. dissolves.
Presence of As.</td><td colspan="2">The pp. does not dissolve.</td></tr>
<tr><td rowspan="2">The pp. does not redissolve.
Presence of Hg (<i>mercuric</i>).</td><td rowspan="2">The pp. becomes white (owing to formation of white insoluble $PbSO_4$).
Presence of Pb.</td><td colspan="2">The pp. dissolves. To separate portions of original solution add—</td><td rowspan="2">An <i>orange</i> pp. indicates presence of Sb.</td><td rowspan="2">A <i>brown</i> or <i>yellow</i> pp. indicates presence of Sn, Stannous (<i>brown</i>), Stannic (<i>yellow</i>).</td></tr>
<tr><td>A little AmCl and an excess of water.
A <i>white</i> pp. indicates presence of Bi.</td><td>AmHO in excess.
A <i>blue</i> solution indicates presence of Cu.</td></tr>
<tr><td colspan="8">Confirm by special tests on pp. 33 to 42.</td></tr>
</table>

TABLE III.

<table>
<tr><th colspan="3">EXAMINATION FOR THE METALS IN GROUP III.</th></tr>
<tr><td colspan="3">The pp is</td></tr>
<tr><td>*Reddish-brown.*
Presence of
Fe.
Test original solution for Fe″ or Fe‴ by K_4FeCy_6, $K_6Fe_2Cy_{12}$, and (KCyS and $HgCl_2$).</td><td>*Dark green.*
Presence of
Cr.</td><td>*White gelatinous.*
Presence of
Al.</td></tr>
<tr><td colspan="3">Confirm by special tests on pp. 43 to 46.</td></tr>
<tr><td colspan="3">*Note.*—Manganese, if present, is usually partially pptd. in this group. *Manganous* hydrate is white and its formation is prevented by the presence of ammonium chloride (hence use of this reagent in Group III.), but, on exposure to air, the solution absorbs oxygen and a hydrated *manganic* oxide. $Mn_2O_2(OH)_2 = Mn_2O_3H_2O$ is slowly thrown down in the form of a brown precipitate. If, therefore, the solution, after addition of AmCl and AmHo, remains clear for a short time, and then a pp. of a dark brown colour begins to form, manganese may be suspected, and the addition of Am_2S to the turbid liquid will produce the buff-coloured pp. of MnS.</td></tr>
</table>

TABLE IV.

<table>
<tr><th colspan="4">EXAMINATION FOR THE METALS OF GROUP IV.</th></tr>
<tr><td colspan="4">The pp. is</td></tr>
<tr><td colspan="2">*Black.* To a portion of the original solution add NaHO.</td><td rowspan="3">*Buff*
or
flesh-coloured.
Presence of
Mn.</td><td rowspan="3">*White.*
Presence of
Zn.</td></tr>
<tr><td>An *apple-green* pp. indicates presence of
Ni.</td><td>A *bluish-white* pp. indicates presence of
Co.</td></tr>
<tr><td colspan="2">**Ni** and **Co** are readily detected in the Preliminary examination by the bead test.</td></tr>
<tr><td colspan="4">Confirm by special tests on pp. 47 to 50.</td></tr>
</table>

TABLE V.

<table>
<tr><td colspan="3">EXAMINATION FOR THE METALS OF GROUP V.</td></tr>
<tr><td colspan="3">To a portion of the solution, which must not be too acid, add $CaSO_4$.</td></tr>
<tr><td>A *white* pp., produced *immediately*, indicates presence of
Ba.</td><td>A *white* pp., produced *after* gently warming and stirring the solution for *some time*, indicates presence of
Sr.</td><td rowspan="2">*No* pp. indicates presence of
Ca.
Confirm by adding $Am_2C_2O_4$ to a fresh portion of original solution. A *white* pp. of CaC_2O_4 is obtained.</td></tr>
<tr><td colspan="2">**Ba** and **Sr** are readily detected in the Preliminary examination by the flame test.</td></tr>
<tr><td colspan="3">Confirm by special tests on pp. 51 to 53.</td></tr>
</table>

TABLE VI.

EXAMINATION FOR THE METALS OF GROUP VI.	
Heat a portion of original substance (dry, or in solution, better dry) with NaHO or $Ca(OH)_2$ in a test tube. Evolution of NH_3 indicates presence of ...	**NH_4.**
To a portion of original solution add AmCl, AmHO, and Na_2HPO_4, and stir with a glass rod. A *white crystalline* pp. of $MgAmPO_4$ indicates presence of	**Mg.**
Evaporate another portion of solution to dryness. Heat to expel ammonium salts, take up a portion of residue with water, and add (on a watch-glass, see page 55) a few drops of $PtCl_4$. A *yellow crystalline* pp. of $2KCl . PtCl_4$ indicates presence of	**K.**
To another portion of the residue apply flame test. Intense *golden yellow flame* indicates presence of	**Na.**

EXAMINATION FOR ACID.

In testing for acids, the student should be guided by—

(*a*) *The results of the preliminary examination.* In many cases the acid will have been detected in this examination, and it is only necessary to confirm the result in the wet way.

(*b*) *The result of the examination for the base.* The base being now known, and the properties and solubility of the substance being determined, the field of search is often considerably narrowed. If no acid is found, the substance may be an oxide or hydrate, and here again a knowledge of the nature of the base helps to decide whether such a compound is likely to occur.

Preparation of Solution.

1. *The substance is in solution or soluble in water.*

If metals of Groups I. or II. be present, remove them by boiling with a slight excess of pure sodium carbonate, filter, and use the filtrate for the examination for acids. If Groups I. and II. be absent, use the original solution.

2. *The substance is insoluble in water, but soluble in acids.*

Boil a portion of the solid with a strong solution of sodium carbonate, filter, and use the filtrate.

3. *The substance is insoluble.*

Treat as described for the preparation of the solution for the examination for bases, using the aqueous filtrate.

Before adding the reagents, exactly neutralize a portion of the solution with HNO_3, or, if acid, with ammonia. If the solution has been boiled with sodium carbonate, the liquid will be strongly alkaline. In this case, make it distinctly acid with HNO_3, boil to expel CO_2, and then neutralize back again with ammonia.

Observation.	Inference.
1. *To neutral solution add* $BaCl_2$.	
White pp., *insoluble* in HCl.	Presence of H_2SO_4 (or H_2SiF_6).
White pp., *soluble* in HCl.	H_3PO_4, H_2SO_3, H_3BO_3, H_4SiO_4, $H_2S_2O_3$ (pp. dissolves, but S is slowly thrown down), H_2CO_3 (pp. dissolves with effervescence), $H_2C_2O_4$.
Yellow pp.	H_2CrO_4.
2. *To another portion add* $AgNO_3$.	
(*a*) A pp. *soluble* in HNO_3.	
i. White, soluble in ammonia.	HNO_2, $H_2C_2O_4$, H_2CO_3, H_4SiO_4, H_3BO_3.
ii. Yellow.	H_3PO_4, H_3AsO_3.
iii. Red.	H_3AsO_4.
iv. Dark red.	H_2CrO_4.
(*b*) A pp. *insoluble* in HNO_3.	
i. White, soluble in ammonia.	HCl, HCN, HClO.
ii. Yellowish white soluble in ammonia.	HBr.
iii. Yellow, insoluble in ammonia.	HI.
iv. Black, ditto.	H_2S.
v. Orange-red, soluble in ammonia.	H_3FeCy_6.
vi. White, insoluble in ammonia.	H_4FeCy_6.

OBSERVATION.	INFERENCE.
If no pp. is produced by $BaCl_2$ or $AgNO_3$—	
3. *To a portion of solution add a solution of* $FeSO_4$, *and then pour down the side of the test-tube strong* H_2SO_4. See Note, p. 64.	
A *brown ring* formed at junction of fluids indicates	HNO_3.
4. *Heat a small portion of dry substance (or residue after evaporation) in a test-tube with strong* H_2SO_4.	
Evolution of ClO_2 and detonation indicates	$HClO_3$.
If no acid is detected by 1, 2, 3, or 4.	
5. *To a fresh portion of the solution add* Fe_2Cl_6.	
A *red coloration,* not destroyed on boiling, but destroyed by addition of $HgCl_2$, indicates	HCyS.
Deep green or *brown coloration* indicates	$H_6Fe_2Cy_{12}$.
Deep blue pp. indicates ...	H_4FeCy_6.

[H_3AsO_3 and $HAsO_2$ will have been detected in the examination for the base.]

APPENDIX I.

MODEL ANALYSES.

1. WHITE CRYSTALLINE SUBSTANCE. SOLUBLE IN WATER. NEUTRAL.

PRELIMINARY EXAMINATION.

Experiment.	*Observation.*	*Inference.*
Heated in dry bulb tube.	Fuses.	Alkaline salt.
,, with HCl.	SO_2 evolved, and solution yellow with separated sulphur.	$H_2S_2O_3$.
,, ,, H_2SO_4.	H_2S, and finally SO_2 evolved, sulphur [yellow] separated.	,, [H_2S].
,, on charcoal with Na_2CO_3.	Residue gives black stain on silver coin.	Compounds of S.
,, ,, platinum-wire.	Yellow flame.	Na.

WET EXAMINATION FOR BASE.

Added HCl.	SO_2 evolved on heating, and S precipitated.	[$H_2S_2O_3$]. Absence of Grp. I.
,, SH_2.	No pp.	Absence of Grp. II.
,, AmCl, AmHO.	,, ,,	,, ,, ,, III.
,, ,, ,, and Am_2S.	,, ,,	,, ,, ,, IV.
,, ,, ,, ,, Am_2CO_3.	,, ,,	,, ,, ,, V.
,, ,, ,, ,, Na_2HPO_4.	,, ,,	,, ,, Mg.
Boiled solution with NaHO.	NH_3 *not* given off.	,, ,, NH_4.
Added $PtCl_4$ and alcohol.	No pp.	,, ,, K.

Base present must be **Na,** confirmed by Preliminary Examination.

EXAMINATION FOR ACID.

Added $BaCl_2$.	White pp. soluble in HCl, solution slowly precipitates sulphur.	$H_2S_2O_3$.
,, $AgNO_3$.	White pp. soluble in excess of solution; on adding excess of $AgNO_3$ and gently heating, pp. becomes black with Ag_2S.	**$H_2S_2O_3$.**

Confirmed by Preliminary Examination.

Base, NaHO. **Acid, $H_2S_2O_3$.**

[**Sodium thiosulphate, $Na_2S_2O_3$.**]

2. WHITE CRYSTALLINE SOLID. SOLUBLE IN WATER. NEUTRAL.

Preliminary Examination.

Experiment.	*Observation.*	*Inference.*
Heated in dry bulb tube.	Deflagrates, brown fumes.	Nitrate of heavy-metal.
,, with HCl.	—	—
,, ,, H_2SO_4.	Brown nitrous fumes.	HNO_3.
,, on charcoal.	Deflagrates.	Nitrate, chlorate.
,, ,, platinum-wire.	Crimson flame.	Sr.

Examination for Base.

Added HCl.	No pp.	Absence of Grp. I.
,, H_2S to same solution.	,, ,,	,, ,, ,, II.
,, AmCl and AmHO to another portion.	,, ,,	,, ,, ,, III.
,, AmCl and Am_2S to same.	,, ,,	,, ,, ,, IV.
,, AmCl, AmHO, and Am_2CO_3 to another portion.	White pp.	Presence of Grp. V.
,, $CaSO_4$ sol.	White pp. after a few minutes.	Sr.

Base is **Strontium**, confirmed by Flame Test.

Examination for Acid.

To aqueous sol. added $BaCl_2$.	No pp.	Absence of H_2SO_4, H_3PO_4, &c.
,, ,, ,, ,, $AgNO_3$.	,, ,,	,, ,, HCl, HBr, &c.
Added $FeSO_4$, filtered off white pp. ($SrSO_4$), and then added strong H_2SO_4.	Brown ring.	HNO_3.

Confirmed by Preliminary Tests.

Base, SrO. **Acid, HNO_3.**

[**Strontium Nitrate, $Sr(NO_3)_2$.**]

3. WHITE AMORPHOUS POWDER. SLIGHTLY SOLUBLE IN WATER. NEUTRAL.

PRELIMINARY EXAMINATION.

Experiment.	*Observation.*	*Inference.*
Heated in dry bulb tube.	——	——
,, with HCl.	——	——
,, ,, H_2SO_4.	——	——
,, on charcoal with Na_2CO_3.	——	——
Placed residue on silver coin.	Dark stain on coin.	Sulphur compounds.
Heated on charcoal.	——	——
,, ,, ,, with $Co(NO_3)_2$.	——	——
,, ,, platinum-wire.	Red flame (traces of Sr flame).	Ca, Sr.
WET EXAMINATION FOR BASE.		
Added HCl.	No pp.	Absence of Grp. I.
,, SH_2.	,, ,,	,, ,, ,, II.
,, AmCl, AmHO.	,, ,,	,, ,, ,, III.
,, ,, ,, Am_2S.	,, ,,	,, ,, ,, IV.
,, ,, ,, Am_2CO_3.	White pp.	Ba, Sr, Ca.
,, $CaSO_4$.	No pp.	**Ca.**
,, $Am_2C_2O_4$.	White pp.	**Ca.**
EXAMINATION FOR ACID.		
Added $BaCl_2$.	White pp., insoluble in HCl.	**H_2SO_4.**
	Confirmed by Preliminary Examination.	
Base, CaO.		**Acid, H_2SO_4.**

[Calcium Sulphate, $CaSO_4$.]

4. WHITE AMORPHOUS POWDER. INSOLUBLE IN WATER. SOLUBLE IN HCl.

PRELIMINARY EXAMINATION.

Experiment.	*Observation.*	*Inference.*
Heated in dry bulb tube.	Becomes yellow-white when cool.	Zn.
" with HCl.	Dissolves with slight effervescence.	CO_2 (*traces*).
" " H_2SO_4.	——	——
" on charcoal with Na_2CO_3.	Yellow when hot, white when cold.	Zn.
" " " " $Co(NO_3)_2$.	Heated mass becomes green.	Zn.
" " platinum-wire.	Reddish flame.	Trace Ca (?).
WET EXAMINATION FOR BASE.		
Added HCl (as solvent).	——	Absence of Grp. I.
" SH_2.	——	" " " II.
" AmCl, AmHO.	——	" " " III.
" " " Am_2S.	White pp., insoluble in acetic acid.	**Zn.**
" NaHO.	White gelatinous pp., soluble in excess.	"
EXAMINATION FOR ACID.		
Boiled portion of solid substance with Na_2CO_3, filtered, neutralized filtrate with HNO_3, and added		
$BaCl_2$.	——	——
$AgNO_3$.	——	——
Fe_2Cl_6.	——	——
Substance is *insoluble* in water …	… … … …	Absence of HNO_3 and $HClO_3$.
[No acid present.]		

Base, ZnO. **[Zinc Oxide.]**

5. WHITE CRYSTALLINE SUBSTANCE. SOLUBLE IN WATER. NEUTRAL.

PRELIMINARY EXAMINATION.

Experiment.	*Observation.*	*Inference.*
Heated in dry bulb tube.	Water of crystallization evolved. Residue yellow to brown when hot.	Pb, Zn, Cd.
,, with HCl.	—	—
,, ,, H_2SO_4.	Brown fumes (Br).	HBr.
,, on charcoal with Na_2CO_3.	Dark brown infusible mass, reddish brown incrustation.	Cd.
,, ,, ,, ,, $Co(NO_3)_2$.	—	—
,, ,, platinum-wire.	—	—

WET EXAMINATION FOR BASE.

Experiment.	*Observation.*	*Inference.*
Added HCl.	No pp.	Absence of Grp. I.
,, H_2S.	Yellow pp., insoluble in Am_2S and Am_2CO_3, but soluble in HNO_3.	**Cd.**
,, AmHO.	White pp., soluble in excess.	,,

EXAMINATION FOR ACID.

Experiment.	*Observation.*	*Inference.*
Added $BaCl_2$.	—	—
,, $AgNO_3$.	Yellowish white pp., difficultly soluble in AmHO.	**HBr.**
Heated substance with MnO_2 and H_2SO_4.	Brown vapour of Br.	,,
Chlorine water added, and solution shaken up with CS_2.	Brown solution of Br. in CS_2 sinks to the bottom.	,,

Base, CdO. **Acid, HBr.**

[Cadmium Bromide, $CdBr_2$.]

6. WHITE CRYSTALLINE SUBSTANCE. SOLUBLE IN WATER. NEUTRAL.

	PRELIMINARY EXAMINATION.	
Experiment.	*Observation.*	*Inference.*
Heated in a dry bulb tube.	Brown oxides of N evolved.	Nitrate of heavy metals.
„ with HCl.	[White insoluble substance formed on addition of acid; dissolves on heating.]	[Grp. I. (Pb)].
„ „ H_2SO_4.	Oxides of N.	HNO_3.
„ „ Na_2CO_3.	Deflagrates: metallic malleable bead which marks paper, blue flame.	Pb.
„ on platinum-wire.	Blue flame.	Pb, As, Sb.
	WET EXAMINATION FOR BASE.	
Added HCl.	White pp.: dissolves on boiling: SH_2 added to solution thus obtained: *reddish-brown* (colour due to excess of HCl), finally black pp. is obtained.	**Pb.**
„ KI.	Yellow pp.	„
„ K_2CrO_4.	„	„
	EXAMINATION FOR ACID.	
Boiled with Na_2CO_3 to remove Pb, neutralized with HNO_3, and added $BaCl_2$.	No pp.	
Added $AgNO_3$.	„	
Pptd Pb from sol. with Na_2CO_3, filtered, acidified with H_2SO_4, and added $FeSO_4$, and H_2SO_4.	Brown ring formed.	HNO_3.
$FeSO_4$ pptates Pb as $PbSO_4$. See p. 64, Note.		
Base, PbO.	[**Lead Nitrate, $Pb(NO_3)_2$**].	**Acid, HNO_3.**

7. WHITE AMORPHOUS POWDER. INSOLUBLE IN WATER AND ACIDS.

PRELIMINARY EXAMINATION.

Experiment.	*Observation.*	*Inference.*
Heated in dry bulb tube.	—	—
" with HCl.	—	—
" " H_2SO_4.	—	—
" on charcoal with Na_2CO_3.	Residue stains silver coin.	S compounds.
" on platinum-wire.	Yellowish-green flame.	Ba.

WET EXAMINATION FOR BASE.

Fused original substance in porcelain crucible with ten times its weight of Na_2CO_3. Boiled fused mass in water, filtered, dissolved residue in HCl and examined for Base.

Added SH_2.	—	Absence of Grp. II.
" AmCl, AmHO.	—	" " III.
" " " Am_2S.	—	" " IV.
" " " Am_2CO_3.	White pp.	Ba, Sr, Ca.
" $CaSO_4$.	Immediate white pp.	**Ba.**

Confirmed by Preliminary Examination.

EXAMINATION OF FILTRATE FOR ACID.

Neutralized with HNO_3.	—	—
Added $BaCl_2$.	White pp. insoluble in HCl.	H_2SO_4.

Confirmed by Preliminary Examination and by insolubility of salt.

Base, BaO. **[Barium Sulphate, $BaSO_4$].** **Acid, H_2SO_4.**

8. ORANGE-RED, AMORPHOUS POWDER, INSOLUBLE IN WATER, SOLUBLE IN STRONG HCl, WITH DECOMPOSITION.

PRELIMINARY EXAMINATION.

Experiment.	*Observation.*	*Inference.*
Heated in dry tube.	—	—
,, with HCl.	Smell of H_2S { Confirmed by blackening of lead }	A sulphide.
,, H_2SO_4.	,, ,, { acetate paper. }	,,
,, on charcoal with Na_2CO_3.	Residue gives black stain on silver coin.	Sulphur compound.
,, on platinum wire.	Violet flame.	K, Pb, As, Sb, Bi.

EXAMINATION FOR BASES.

Added HCl (to dissolve).	Soluble in the concentrated acid with evolution [of H_2S.	Absence of Grp. I.
,, H_2S to diluted solution.	Orange-red pp.	Sb.
Treated pp. with Am_2S.	Soluble.	,,
,, ,, ,, Am_2CO_3.	Insoluble.	,,
In Marsh's apparatus.	Black stain, insoluble in NaClO.	,,

EXAMINATION FOR ACID.

The Preliminary experiments conclusively show........................	Presence of sulphide.

Base, Sb_2O_3.	**Antimony Sulphide, Sb_2S_3.**	**Acid, H_2S.**

APPENDIX II.

TABLE SHOWING THE SOLUBILITY OF THE MORE COMMONLY OCCURRING SALTS.

NAME.	SOLUBLE IN WATER.	SOLUBLE IN, OR DECOMPOSED BY, ACIDS.	INSOLUBLE.
Carbonates.	Acid carbonates of Ba, Sr, Pb, Mn, Mg, Fe'', and Ca, and alkaline carbonates.	All carbonates are decomposed with evolution of CO_2.	
Sulphates.	Most sulphates. (Ag_2SO_4 is difficultly soluble.)	Some *basic* sulphates.	Sulphates of Pb, Ba, Sr and Ca, and red chromic sulphate.
Nitrates.	Most nitrates (a few *basic* nitrates excepted).	A few basic nitrates which are readily soluble in dilute HNO_3.	
Chlorides.	Most chlorides.	A few oxychlorides. ($BiOCl$ and $SbOCl$.)	Chlorides of Pb, $'Hg'_2$, Ag, and Cu_2Cl_2.
Bromides.	Most bromides.	Cu_2Br_2 in HCl.	Bromides of $'Hg'_2$, Ag, and Pb.
Iodides.	Most iodides.	HgI_2, Hg_2I_2 $(= HgI_2 + Hg)$, PbI_2, Cu_2I_2, decomposed by HNO_3 with evolution of I.	Iodides of Ag, $'Hg'$, Hg'', Pb, and $'Cu'$.
Chlorates.	All chlorates.		
Sulphides.	Alkaline and alkaline earthy sulphides. MgS and CaS *very slightly* soluble.	All other sulphides are decomposed by HCl (H_2S being evolved), or by HNO_3, or aqua regia, with separation of S.	
Phosphates.	Alkaline phosphates only.	Remainder are soluble.	
Chromates.	Alkaline chromates and chromates of Mg, Zn, Ca, Sr, Fe^{iv}, and Cu.	Chromates insoluble in water dissolve in acids (evolving chlorine in concentrated HCl).	Ignited lead chromate.

APPENDIX III.

SUBSTANCES GIVEN FOR ANALYSIS AT LOND. INTER. SC. AND PREL. SCI. EXAMS.

Two solutions were given to each candidate for analysis, each solution containing one of the following substances :—

1881.

Potassic Carbonate, Sodic Chloride, Calcium Chloride, Ammonic Chloride, Manganous Sulphate, Barium Chloride, Zinc Sulphate, Ferric Chloride, Lead Nitrate, Ferrous Sulphate, Antimonious Chloride, Magnesic Chloride, Bismuth Nitrate, Boracic Acid, Mercurous Nitrate, Nickel Sulphate, Alum, Stannous Chloride.

1882.

Potassic Carbonate, Sodic Chloride, Calcium Chloride, Ammonic Chloride, Manganous Sulphate, Barium Chloride, Zinc Sulphate, Ferric Chloride, Lead Nitrate, Ferrous Sulphate, Antimonious Chloride, Magnesic Chloride, Bismuth Nitrate, Boracic Acid, Mercurous Nitrate, Nickel Sulphate, Alum, Stannous Chloride.

1883.

Cadmium Sulphate, Barium Nitrate, Bismuth Nitrate, Sodium Phosphate, Ferrous Sulphate, Potassium Nitrate.

1884.

Sodium Thiosulphate, Mercurous Nitrate, Cadmium Sulphate, Potassium Nitrite, Potassium Bromide, Silver Nitrate.

1885.

Mercuric Chloride, Ammonium Chloride, Potassium Iodide, Manganese Chloride, Borax, Potassium Bromide, Zinc Sulphate, Potassium Chlorate, Ammonium Phosphate, Sodium Carbonate, Barium Chloride, Manganese Sulphate, Sodium Chloride, Nickel Sulphate, Silver Nitrate, Calcium Chloride, Sodium Thiosulphate, Sodium Arsenite, Bismuth Nitrate, Mercurous Nitrate, Cadmium Nitrate.

1886.

Zinc Sulphate, Potassium Bromide, Ammonium Chloride, Barium Nitrate, Sodium Phosphate, Mercuric Chloride, Manganese Chloride, Potassium Carbonate, Magnesium Sulphate, Ammonium Nitrate, Nickel Nitrate, Potassium Chloride, Ammonium Chromate, and Cobalt Nitrate.

1887.

Mercuric Chloride, Manganese Chloride, Ferrous Sulphate, Zinc Sulphate, Lead Nitrate, Barium Nitrate and Chloride, Calcium Chloride, Sodium Phosphate and Thiosulphate, Potassium Chloride, Bromide, Iodide, Sulphate, and Carbonate, Ammonium Chloride and Phosphate, Magnesium Sulphate.

JAN. PREL. SCI.—Sodium Carbonate, Zinc Sulphate, Ammonium Chloride, Manganese Sulphate, Sodium Nitrate, Manganese Chloride.

1888.

Mercuric Chloride, Manganese Sulphate, Manganese Chloride, Potassium Chloride, Sodium Phosphate, Nickel Nitrate and Chloride, Bismuth Nitrate, Borax, Cadmium, Nitrate and Sulphate, Potassium Chloride, Ammonium Nitrate, Potassium Chromate and Carbonate, Cobalt Nitrate and Ammonium Sulphate.

JAN. PREL. SCI.—Mercuric Chloride, Manganese Chloride, Zinc Sulphate, Ammonium Phosphate.

1889.

Potassium Bromide, Manganese Sulphate, Potassium Carbonate, Lead Nitrate, Ammonium Chloride, Bismuth Nitrate, Boric Acid, Sodium Sulphite, Nickel Chloride, Borax.

JAN. PREL. SCI.—Ammonium Nitrate, Potassium Chloride, Manganese Sulphate, Mercuric Chloride, Zinc Sulphate, Lead Nitrate.

1890.

JAN. PREL. SCI.—Potassium Bromide, Bismuth Nitrate, Aluminium Sulphate, Sodium Arsenite, Zinc Chloride, Barium Chlorate.

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